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IMPROVEMENT OF ERODIBLE SOILS THROUGH USE OF OIL INDUSTRY WASTES

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IMPROVEMENT OF ERODIBLE SOILS THROUGH USE OF OIL INDUSTRY WASTES

by

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and

SOILS AT THE RICHMOUND OILY WASTE RESEARCH SITE
(APPENDIX A)

by

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EXECUTIVE SUMMARY

Since traditional methods of disposing of petroleum production and processing wastes, such as rural road oiling and burial, have lost favour with regulatory agencies and can no longer accommodate the growing waste volumes, there is a current search for alternatives. We propose that agriculture can provide effective solutions for some disposal problems by utilizing the organic carbon-rich wastes to amend nearby fields of marginal productivity. In the Prairie region there are 7.7 million cultivated acres of sandy soils (>50% sand) that are highly susceptible to degradation by wind and water erosion and are low in organic matter and fertility. These soils would benefit from the improvements in structure and humus content that can result from incorporation of oily wastes, particularly petroleum wastes that are typically rich in aromatics as opposed to oily food wastes containing mainly long-chained aliphatics that are rapidly metabolized and almost completely mineralized. In an earlier study near Maidstone, SK a single application of a heavy oil production sludge to a loamy sand to an initial 1-2% soil oil content effected large and lasting increases in soil aggregation plus a 15 t/acre gain of humus via microbial conversion of hydrocarbons. However, cereal production in the early years was severely reduced by the adverse effects from sludge-associated salts.

Consequently, the current field study was conducted using a practically salt-free heavy oil processing sludge on a windy ridge near Richmound, SK to

1. assess the impact of topsoil treatment with oily waste on soil quality and cereal production, and to
2. generate data for regulatory agencies, so they can develop guidelines for safely using suitable oily wastes in the future to improve sandy cropland.

On November 5, 1997, heavy oil processing waste sludge from the Consumers' Co-operative Refineries Limited (CCRL) Upgrader in Regina, SK was applied at three

rates (for 0, 1 and 2% initial soil oil content) together with the first 1/3 of 2 nitrogen and phosphorus fertilizer rates (none and best management) and rotovated into the top four inches of an eroded Hatton loamy sand. All treatments were replicated four times on a total of 96 plots (2m x 12m). Next spring, the remaining 2/3 of fertilizer was applied in two applications for a total of 625 lb N/acre and 215 lb P_2O_5/acre at the initial 1% soil oil content and double at the 2% soil oil content. Then 48 plots were chemfallowed while 24 plots were seeded to Kyle durum and 24 plots to Calibre oats. Due to the effects of late fall and early spring tillage plus a dry winter and very dry summer, crop production was generally poor in 1998 on most plots. In 1999, the 48 previously fallowed plots were seeded to durum and oats and subsequent crop growth was good under more favourable weather conditions. Soil samples for physical, chemical and microbial analyses were collected from all plots within one and 21 days of oily waste incorporation and then each spring and fall until spring 2000. The applied CCRL sludge consisted of 61% water, 31% hydrocarbons and 8% solids (clay). It contained 5000 ppm of polynuclear aromatic hydrocarbons (PAH) but only few volatiles (420 ppm) and salts (280 ppm) or nutrients (70 ppm N, 20 ppm P) except for sulfur (6000 ppm).

In this fragile loamy sand the oily waste served initially as an effective 'glue' for binding the sand grains together. Thus, by early spring 1998 the oiled plots showed no signs of any topsoil loss to wind erosion while the unoiled control plots were littered with rocks and pebbles that became exposed after the surrounding topsoil was blown away by strong Chinook winds. Rotary dry sieving of soils sampled that spring, showed the wind erodible soil fraction (i.e. aggregates <0.84 mm) was reduced by 9% where 1% oil had been applied and by 13% where 2% was the initial oil content. These reductions in susceptibility to wind erosion were maintained relatively unchanged, while the waste oil underwent biodegradation, at least until spring 2000, i.e. 29 months after sludge application. Resistance to erosion by water was boosted even more than dry aggregate stability since wet sieving of the spring 1998 samples indicated increases of 44% and 64% in water stable aggregates where sludge had been applied to a 1% and 2% soil oil content. The large increases in stable aggregation and related improvements in soil structure persisted at least until the spring 2000 sampling. These oil-induced aggregation benefits lead us to suggest that a single sludge application, every 4 to 6 years, should provide good protection against topsoil erosion, even on very sandy land.

Incorporation of the oily waste sludge stimulated not only the oil degrading microorganisms but also most other soil microbes and, even two years later, populations of bacteria and fungi in oiled soils were several fold greater than in controls particularly in plots that were heavily fertilized at the start. Lower soil microbial respiration in those fertilized plots indicated that nutrient supplementation improves the efficiency of bioconverting hydrocarbons to stable soil organic matter, i.e. humus. The initial heavy N and P fertilization of the oiled plots put more nutrients into the topsoil than could be microbially immobilized or physico-chemically absorbed by the sand thus resulting in massive leaching of nitrates and phosphates down to the cereal rooting depth and beyond. However, recent deep coring has shown that no significant amounts of phosphate-P or ammonium-N would migrate so deep as to reach the groundwater. Furthermore, repeated analyses of samples, collected to date, from the six on-site groundwater monitoring wells have failed to detect any PAHs or other oil-contained organics however elevated nitrate-N levels were found in spring 2000.

Plant emergence counts in 1999 indicated no residual phytotoxic effects from the waste oil application on plant density or development of either crop. Yet the soil-incorporated oily wastes exerted an obvious ‘herbicidal’ effect since the cereal stands in oiled plots were practically free of weeds in 1998 and 1999 while stands in the unoiled control plots were generally very weedy. Grain yield was measured on September 2, 1999 by harvesting a 10 m long strip down the middle of each plot with a small combine. Even without the earlier oily waste-related massive fertilization durum yields were significantly increased by sludge application, exceeding check yields by 40% at the 1% initial soil oil content and by 80% at the 2% soil oil content. Residual benefits from that fertilization were still reflected as yield increases, particularly the 160% higher yield at 1% soil oil content. Despite these yield increases protein contents of durum on oiled plots were generally above the 14.5% level of the check plots, thus indicating high grain quality. For corresponding treatments, the oat crop generally yielded better than durum on this sandy soil. Oat yields also were significantly increased where only sludge had been applied thus corroborating the beneficial effects from oil-induced soil improvements for crop production on marginal land. Oat yields were further enhanced by residual effects from initial heavy fertilization resulting in about 50% and 70% more grain harvested from 1% and 2% waste oiled soils than from check plots.

The results of this field study support our claim that some oily wastes can serve as an effective resource to protect and improve sandy cropland for greater grain production and that this agricultural use could become a safe and economical waste disposal alternative for the petroleum industry in western Canada.

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1. INTRODUCTION

With the increasing demand for development of heavy oil deposits in western Canada due to diminishing supplies of light to medium crude oil, more petroleum wastes are being generated particularly with the adoption of enhanced oil recovery (EOR) processes. Although the efficiency of refinery extraction has improved there are still considerable amounts of oily wastes containing non-economically extractable hydrocarbons (HC). These wastes are often stored for long periods of time in tanker cars and tailing ponds and now they can also be pumped into subterranean caverns for storage where possible.

Use and disposal options for these types of wastes have been discussed extensively elsewhere (Biederbeck et al. 1993). Road oiling and landfarming are not necessarily the most desirable options because of contamination of adjacent ecosystems and production of greenhouse gases (CO_2), respectively.

In this study, we propose that agriculture may provide effective solutions for some oily waste disposal problems by utilizing the organic C-rich wastes to amend nearby areas of marginally productive cropland. Cultivated land on the Prairies includes >3 million hectares of sandy soils (>50% sand) that are highly susceptible to degradation by wind and water erosion and are low in organic matter and productivity. Much of this marginal cropland could benefit from improvements in soil structure and humus content that can be realized through incorporation of organic, including petro-type, wastes (Biederbeck et al. 1997). However, as shown in a recent study of application of oily food wastes to agricultural land (Plante and Voroney 1998), oily materials that consist mainly of long-chain aliphatics, such as canola oil, can greatly boost soil microbial biomass and acctivity and do temporarily promote water stable aggregation through the binding action of microbial products and metabolites (Dinel et al. 1992). These oily food wastes are decomposed so rapidly (i.e. within weeks) and mineralized so completely however, that they cannot serve as substrates for humus formation and, in turn, long-term improvement of soil productivity. It is suggested that 'petro-type' oily wastes, inherently rich in aromatics, may serve as very effective source materials for microbial conversion to stable soil organic matter and gradual humification (McGill et al. 1981).

An earlier field study in northwest Saskatchewan found that heavy oil production wastes could be useful for improving erodible, marginal cropland however the Na content of the associated aqueous phase (brine) was very high and thus detrimental to crop growth and therefore all of the benefits from a one-time application of oily waste to agricultural soil were not realized (Biederbeck at al. 1993). Thus we conducted, with support from AFIF, a field experiment on a windy ridge near Richmound, SK which combined a low Na oily waste sludge with a marginal, erodible cropland in need of protection and improvement. The approach of this study was to optimize the microbial bioconversion of HC to humus-type materials so as to reduce greenhouse gas emissions and also effect long-term soil improvements.

This report is a summary of a three year study on the feasibility of using a refinery-generated heavy oil waste sludge for the protection and improvement of a sandy soil, cultivated for cereal crop production in southwestern Saskatchewan. The experimental site was monitored continuously and thoroughly to assess if surface soil quality can be improved without excessive contamination of crops and groundwater with undesirable chemicals; so as to facilitate future development of an environmentally sound and agriculturally more beneficial disposal option for petroleum production and processing wastes.

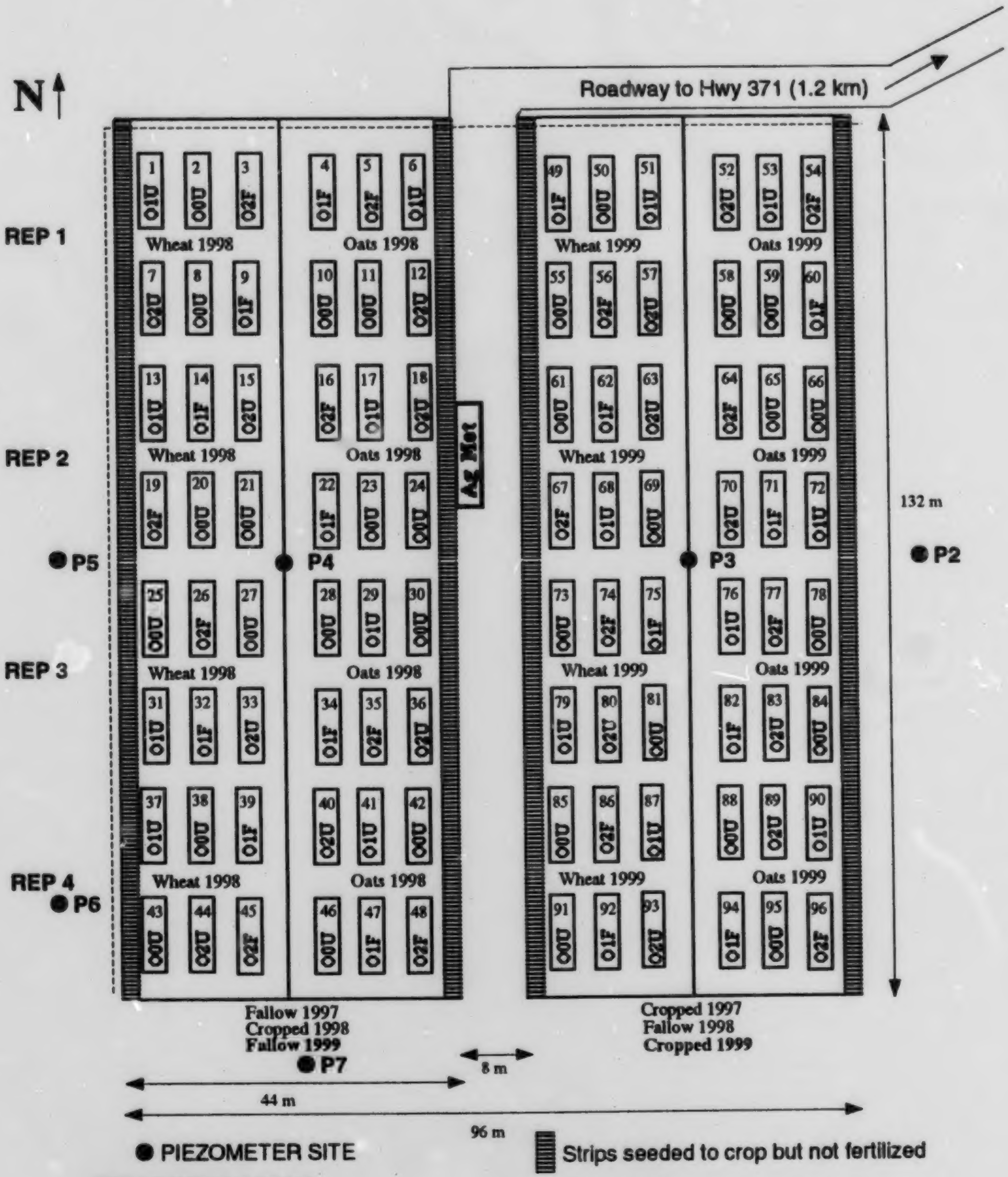
2. STUDY OBJECTIVES

Development of a research database for utilization of oil production and processing wastes as an amendment for erodible, marginal cropland is essential to ensure these wastes do not create environmental problems. Previously, disposal of oil production wastes has been a source of friction between heavy oil producers, regulatory agencies and farmers. Thus this study was conducted to continue the development of an environmentally sound disposal method for heavy oil production wastes to improve marginal soil thus linking the petroleum industry and agriculture in a mutually beneficial manner.

Planning for this study included objectives that determined effects of oily waste application and its interaction with agronomic practices (including crop phase and fertilization) on soil aggregation, bioconversion of hydrocarbons to humus, crop production and monitoring for undesirable effects such as soil or groundwater contamination. To meet all of these objectives a large plot experiment was established at the study site near Richmound, SK (Figure 2.1).

The main objectives of the study at the field site on a windy ridge near Richmound, SK were:

- to assess the impact of soil incorporation of a practically salt-free heavy oil processing sludge on soil and groundwater quality and on crop production, and
- to generate benchmark data needed by regulatory agencies to develop guidelines for future use of suitable HC wastes in an environmentally safe and agronomically beneficial manner on sandy cropland in Saskatchewan and Alberta.


N
Roadway to Hwy 371 (1.2 km)
REP 1
REP 2
REP 3
REP 4
1 O1U
2 O0U
3 O2F
4 O1F
5 O2F
6 O1U
Wheat 1998
Oats 1998
7 O2U
8 O0U
9 O1F
10 O0U
11 O0U
12 O2U
49 O1F
50 O0U
51 O1U
52 O2U
53 O1U
54 O2F
Wheat 1999
Oats 1999
55 O0U
56 O2F
57 O2U
58 O0U
59 O0U
60 O1F
13 O1U
14 O1F
15 O2U
16 O2F
17 O1U
18 O2U
Wheat 1998
Oats 1998
19 O2F
20 O0U
21 O0U
22 O1F
23 O0U
24 O0U
Ag Met
61 O0U
62 O1F
63 O2U
64 O2F
65 O0U
66 O0U
Wheat 1999
Oats 1999
67 O2F
68 O1U
69 O0U
70 O2U
71 O1F
72 O1U
132 m
P5
P4
P3
P2
25 O0U
26 O2F
27 O0U
28 O0U
29 O1U
30 O0U
Wheat 1998
Oats 1998
31 O1U
32 O1F
33 O2U
34 O1F
35 O2F
36 O2U
73 O0U
74 O2F
75 O1F
76 O1U
77 O2F
78 O0U
Wheat 1999
Oats 1999
79 O1U
80 O2U
81 O0U
82 O1F
83 O2U
84 O0U
37 O1U
38 O0U
39 O1F
40 O2U
41 O1U
42 O0U
Wheat 1998
Oats 1998
P6
43 O0U
44 O2U
45 O2F
46 O0U
47 O1F
48 O2F
85 O0U
86 O2F
87 O1U
88 O0U
89 O2U
90 O1U
Wheat 1999
Oats 1999
91 O0U
92 O1F
93 O2U
94 O1F
95 O0U
96 O2F
Fallow 1997
Cropped 1998
Fallow 1999
Cropped 1997
Fallow 1998
Cropped 1999
P7
8 m
44 m
96 m
● PIEZOMETER SITE
▥ Strips seeded to crop but not fertilized

TREATMENT CODES:

O0 - no oil added

O1 - 1% oil added to top 10 cm soil (w/w)

O2 - 2% oil added to top 10 cm soil (w/w)

U - unfertilized for oil degradation

F - fertilized for oil degradation

AgMet site - has soil temperature probes in plots 18, 24, 61 & 67 at 5 cm depth

NOTE: Not to scale. The plots are 2 m x 12 m with 4 m between plots in the E-W direction and 4 m between plots in the N-S direction.

Figure 2.1. Plot arrangement for experiment on incorporation of oily waste into Hatton loamy sand near Richmound, SK.

3. PROJECT SITE SELECTION AND DESCRIPTION

In July 1997, Dr. Volkmar Biederbeck and Mr. Keith Hanson from Agriculture and Agri-Food Canada in Swift Current and Mr. Terry Fonstad from the Dept. of Agricultural and Bioresource Engineering of the University of Saskatchewan in Saskatoon made a field trip to Richmound, SK to meet with an interested local producer/landowner, Mr. Ben Kambeitz and determine the most appropriate site for the field study. Mr. Kambeitz had conducted some earlier independent trials on his land utilizing petroleum wastes to reduce erosion on his land and was eager to continue this work with sound scientific support and the approval of regulatory agencies. After evaluating a few fields, a relatively flat area at the top of a windy ridge was chosen as the best site for the trial. Surface soil and bulk density samples were taken for physical and chemical analyses to ensure the site met all test requirements.

The site selected is located in the southeast quarter of Section 20 in Township 17 and Range 27, west of the third Meridian in the R.M. of Fox Valley No. 171. The site is approximately 6 km southeast of Richmound, SK and can be reached by driving from the eastern edge of Richmound 5.6 km east on Highway 371, then 1.0 km south on a well maintained road allowance and finally 0.2 km southwest on a field trail. The experimental area was located several hundred meters from the nearest surface water (a slough) and at a point in the field where a division between cropped and fallow areas would allow a continuation of conventional cropping phases after oily waste addition. The land slopes away from the site toward east, south and west directions (Figure 3.1). The soil in the study area is light textured belonging to the Hatton Soil Association. A complete description of the soils at the site can be found in an attached report (Appendix A). This site was considered most suitable because:

(i) the landowner was very interested in the project and willing to cooperate in any way possible;

(ii) the site had both fallow and cropped phases in the 1997 crop year and would fit well into the experimental plan of the study;

(iii) the site was highly susceptible to erosion, especially wind erosion, and would provide a good test of the technology for 'erosion-proofing' the soil;

(iv) the field had previously been managed as a chem-fallow cereal rotation for several years using good agronomic practices; and

(v) the site was easily accessible throughout most of the year while protected against intruders or vandals with its location being well off the highway.

To determine the soil characteristics and check the homogeneity across the study site 5 cm wide cores were taken hydraulically to a 120 cm depth in 28 interplot locations in the spring of 1998 (i.e. just prior to application of the second amount of fertilizer for best-management treatments). These 'benchmark' samples were analyzed for physical and chemical properties. Each core was separated into five depth segments (0-10, 10-30, 30-60, 60-90 and 90-120 cm) and the mean values for the top two depths, i.e. 0-10 and 10-30 cm, are shown in Table 3.1.

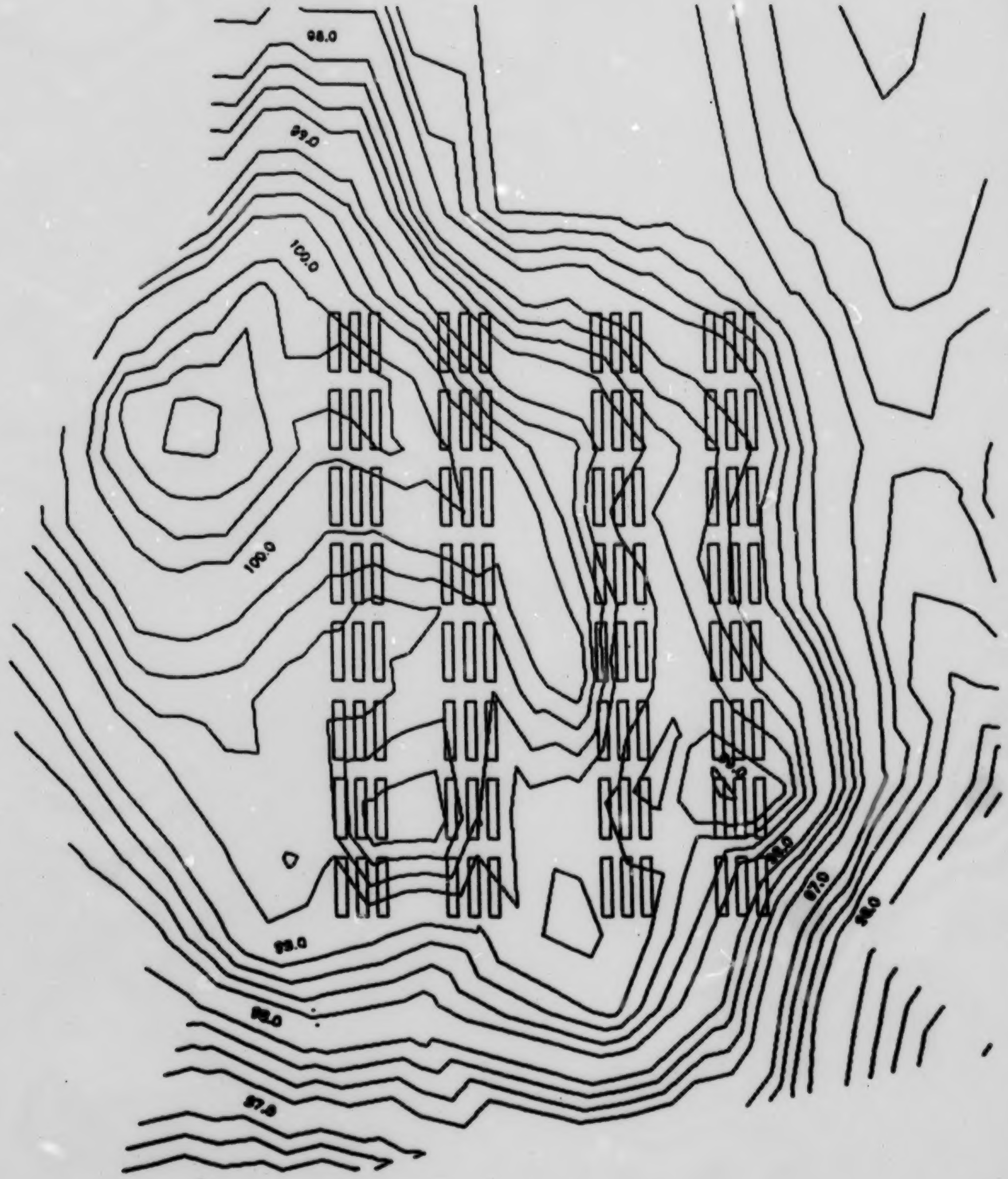

98.0
99.0
100.0
100.0
98.0
97.0
96.0
99.0
98.0
97.0

Figure 3.1 Topography of Hatton loamy sand at 1.3 ha oily waste study site in SE 20-17-27 W3M near Richmound, SK (Contour lines run at 0.25m intervals).

Table 3.1 Major physical and chemical properties of the Hatton loamy sand at the field study site near Richmound, SK in April 1998.

	Depth, cm	
Soil Property[†]	0-10	10-30
Texture:		
Sand, %	89.0	87.3
Silt, %	6.0	5.9
Clay, %	5.0	6.8
Textural class	loamy sand	loamy sand
pH[‡]	7.6	nd
Electrical conductivity, dS/m	1.1	nd
Bulk density, Mg/m3	1.24	1.36
Water content:		
At field capacity (0.033 Mpa), %	7.7	8.5
At permanent wilting point (1.5 Mpa), %	3.8	4.0
Total C, %	1.28	1.24
Total N, %	0.079	0.054
Organic C, %	0.78	0.52
C/N ratio	9.9	9.6

[†] Mean from analyses of 28 interplot soil cores.

[‡] In saturation extract.

nd = not determined.

4. FIELD EXPERIMENTS

4.1 EXPERIMENTAL DESIGN

In early November 1997 a plot experiment was established on a 1.3 ha area of Hatton loamy sand (Chernozemic Brown soil) located in the SE quarter of Section 20, Tp 17, Rge 27, W3M in the RM of Fox Valley No. 171, 6 km southeast of Richmound, Saskatchewan. The experiment was run as a fallow-crop rotation with two crops (i.e. oats and durum), three oily waste sludge rates [0, 1, and 2% (wt/wt) in soil], two fertilizer rates (i.e. none and best management), and four replications of each treatment for a total of 96 plots. The site was located at a point in the field where half was cropped in 1997 and half was fallow. The oily waste sludge was applied on November 5, 1997 at three sludge application rates (O0 = none, O1 = 41000, and O2 = 82000 litres/ha) which were used trying to reach targeted waste loadings of 0%, 1.0% and 2.0% oil in the top 10 cm of soil (wt/wt), respectively. These targets were based on analyses received from Consumer's Co-operative Refinery Limited (CCRL) of a composition of 31% oil + 61% water + 8% solids in the sludge used from the Heavy Oil Upgrader facility of CCRL located in Regina, Saskatchewan.

The experimental design was a Randomized Complete Block. Samplings of various types were conducted at different times prior to and after the establishment of the experiments. All the data was statistically analyzed using models in JMP (SAS Institute, Gary, NC) with single degree-of-freedom contrasts for comparisons of selected variables.

4.2 APPLICATION OF OILY WASTE SLUDGE

Application and incorporation of the oily waste sludge to a 10 cm depth was achieved on November 5, 1997 by using an applicator-rotovator (a modified rotovator), pulled by a tractor and connected through its Bowie pump and 3" hose to a vacuum truck running alongside (Figure 4.1). The vacuum truck had previously been filled with oily waste sludge from a tanker truck that had transported the sludge from Regina to the field. The applicator-rotovator (Howard Rotovator HR20) was modified by Fabro Ltd. in Swift Current, Saskatchewan according to instructions from F.B. Dyck, machine design engineer at SPARC. Later, it was revised at SPARC (Engen 1986) because of problems encountered during application of oily waste sludge in a previous experiment. A single pass was required to cover the area of each field plot. To ensure the O2 plots received twice the amount of oily waste sludge as the O1 plots, two passes over the same plot area were done. All unoiled control plots were also roto-tilled to a depth of 10 cm with the applicator-rotovator prior to any soil oiling.

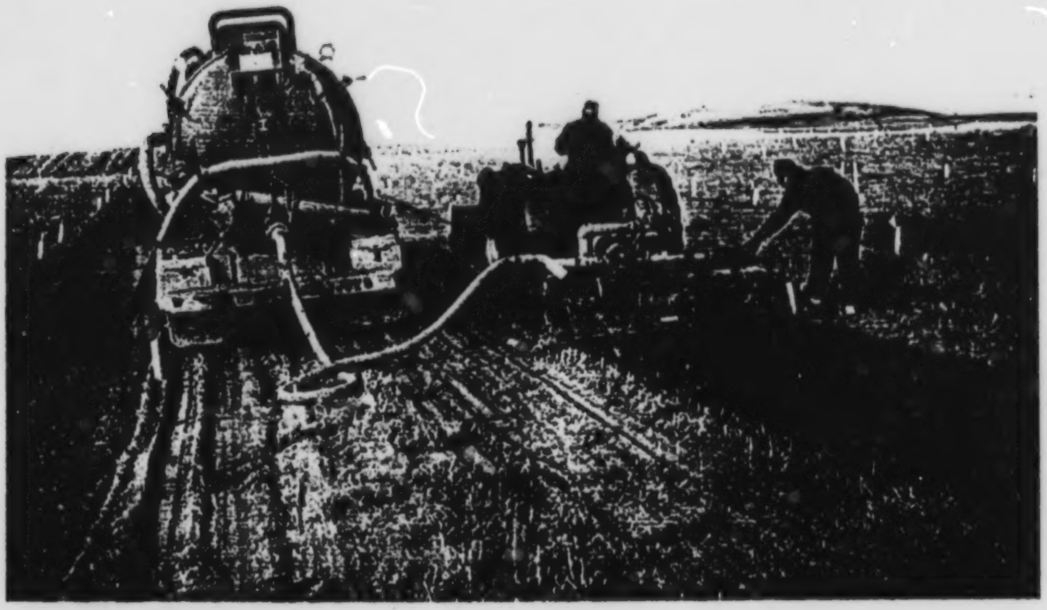


Figure 4.1 Application and soil incorporation of oily waste sludge in November of 1997 at study site near Richmound, SK.

4.3 AGRONOMIC PRACTICES

4.3.1 Fertilization

Fertilization on those plots designated to receive fertilizer consisted of addition and incorporation of nitrogen (N) and phosphorus (P) only because of the high sulphur (S) content in the oily waste sludge itself (over 6000 ppm). The ratio between the N and P was 100:15 and this is similar to the ratio found in stable soil organic matter. The nutrient ratio and amounts of fertilizers were selected to optimize the microbial conversion of the hydrocarbons to soil humus-type materials rather than to maximize oil degradation and CO_2 evolution which would pollute the atmosphere with greenhouse-type gases and could effect a possible net loss of soil organic matter.

The fertilizer was applied in three equal portions to the plots receiving the fertilizer treatments. The initial fertilizer application was conducted one day prior to the oily waste incorporation and was spread onto the soil surface with a Gandy fertilizer broadcaster, which was pulled manually throughout the plots. During the application and incorporation of the oily waste sludge, the initial fertilizer application was incorporated to a depth of 10 cm. The second application was in early April 1998 after soil sampling and the third just prior to seeding of the oats and durum on the west half of the plot area. The second application was broadcast onto the soil surface and left unincorporated. The third application was incorporated with the seeding activity to a depth of 3.5 cm on the west half and incorporated with tine harrows to a depth of 3.5 cm on the unseeded, east half of the plot area.

The oil-related fertilizer rates were as follows: U = unfertilized (none); F = fertilized (best-management). Best-management or F treatment consisted of two different rates depending on the targeted oil content of the soil. F for 1% oil (or O1F) plots was 700 kg N/ha + 105 kg P/ha and for 2% oil (or O2F) plots was 1400 kg N/ha + 210 kg

P/ha. Each of the three applications were divided evenly into 235 kg N/ha + 35 kg P/ha for the O1F and double that for the O2F plots.

4.3.2 Seeding and Harvesting

On April 16, 1998, the roadway was seeded to winter wheat, cv. Norstar, and the west half of the plot area seeded to blocks of durum, cv. Kyle, and oats, cv. Calibre. The blocks seeded to each crop type included the plots and the interplot area. The seeding rates were as follows: Norstar winter wheat – 73 kg/ha (on roadways), Calibre oats – 52 kg/ha, and Kyle durum – 85 kg/ha. The seeded areas were fertilized for crop production with ammonium sulphate and urea at the following rates: 21-0-0-24 at 83.3 kg/ha and 46-0-0 at 70.7 kg/ha resulting in 50 kg N/ha and 20 kg S/ha. The soil contained sufficient available P to supply the crop. The seeding was done using a disc drill with 7" row spacing by Dean James, Soil Fertility technician at SPARC.

Due to very poor crop emergence and high weed density, it was decided by late May to spray out the area with Round-Up and reseed the crops. On May 27, 1998, Round-Up herbicide was sprayed on the cropped and fallow areas at 1 l/ac. Three hours after herbicide application, the crops were reseeded using the same rates as described previously. No fertilizer was applied during this seeding operation.

On August 17th and 31st, after harvest sampling, the site was cleared mechanically with a Wintersteiger harvester operated by Dean James. The variability in maturity of the crops required two dates for harvest completion.

On May 4, 1999, the roadway was seeded again to Norstar winter wheat and the east half of the plot area into blocks of durum, cv. Kyle, and oats, cv. Calibre. The seeding rates were the same as those used in 1998 as was the fertilizer applied at seeding. The seeding was done with the same equipment as the 1998 seeding and was operated by

Dean Klassen of SPARC. The entire cropped area was harvested after sampling on September 2, 1999, using a Wintersteiger harvester.

4.3.3 Tillage and Weed Control Practices

In August 1997, Ben Kambeitz, the producer-owner of the site did a mechanized rock picking operation over the area of the proposed field site that was fallowed in 1997. All other tillage/soil disturbance operations are those related to oil and fertilizer incorporation which were described previously.

On May 27, 1998, the cropped and fallow areas of the field site were sprayed with Round-Up (glyphosate acid) at 0.88 kg a.i./ha by Dean James for general weed and crop control after the decision was made to reseed the site. On June 22, 1998, the cropped and fallow areas were sprayed with Pardner (bromoxynil) at 0.28 kg a.i./ha and the fallow area also with Round-Up at 0.44 kg a.i./ha.

On June 1, 1999, spot spraying of wild oats in the durum stand was conducted by Keith Hanson using Hoegrass 284 at 0.79 kg a.i./ha. The spot spraying was concentrated in the north end of the durum block. An application of Buctril M (bromoxynil and MCPA ester) was applied at 0.55 kg a.i./ha to the entire cropped area and Achieve DG (tralkoxydim) at 0.4 kg a.i./ha to the durum block on June 14, 1999 by Dean James.

4.4 SOIL AND PLANT SAMPLING

4.4.1 Collection of Soil Samples

To meaningfully assess the extent and persistence of the changes in soil physical, chemical and biological properties effected by incorporation of oily waste sludge into Hatton loamy sand and to monitor the biodegradation and bioconversion of oily wastes and its interaction with agronomic practices and crop production, it was necessary to collect different types and sizes of soil samples at various intervals for the wide variety of analyses that were routinely performed on the soil. Consequently, the following methods of soil sampling were used before and during the two and one half years after oily waste sludge application at the field site near Richmound, SK.

(i) To determine the soil characteristics and homogeneity across the project site two 1.8 cm diameter cores to 120 cm depth were taken hydraulically in each of the 96 plots with a truck-mounted Giddings punch prior to oily waste sludge application. Each core was separated into depth segments of 0-10, 10-30, 30-60, 60-90 and 90-120 cm and each of the five depth segments was analyzed for NO_3-N, NH_4-N, PO_4-P and SO_4-S. The 0-10 and 10-30 cm segments of these initial benchmark samples were also analyzed for total N and total and organic C.

(ii) A second set of samples to determine physical soil characteristics and site homogeneity were taken at 28 equidistant interplot points across the study site using 5 cm cores to 120 cm depth taken hydraulically using a truck-mounted Giddings punch. These cores were segmented as described previously and were analyzed for texture, pH, EC and moisture at 0.3 and 15 bar suction. The 0-10 and 10-30 cm segments of these samples were also analyzed for total N and total and organic C.

(iii) To measure changes in soil physical and chemical properties, down the soil profile, due to incorporation and biodegradation of oily wastes, two hydraulic core samplings to 120 cm depth were taken in each plot in fall of 1998 and 1999. Each of the five segments were separated and then composited within each depth segment into one sample for each plot. The 0-10 cm depth segment was supplemented by a third sampling if required to ensure having enough soil for subsequent analyses. Each segment sample was analyzed for NO_3-N, NH_4-N, PO_4-P and SO_4-S. . The 0-10 and 10-30 cm segments of these samples were also analyzed for total N and organic C.

(iv) To measure the extent of biodegradation of soil-incorporated oily waste and to assess changes in soil microbial populations and activities, surface slices 15 cm x 15 cm in area and to a 10 cm depth were taken by trowel within each plot. These samples were collected within 24 h of oily waste application/incorporation, three weeks after and then each spring and fall in 1998 and 1999 for determination of residual oil content and for various microbiological analyses.

(v) To assess changes in a major phase of soil structure, due to incorporation and biodegradation of oily wastes, surface soil was collected periodically to facilitate measurement of the state of primary and secondary soil aggregation. For determination of the size distribution of secondary or field aggregates by rotary sieving, i.e. a measure of the soil's susceptibility to wind erosion, two side-by-side 0-10 cm deep cores were taken with a bulb planter at six different locations in the plot and then combined in 2 litre milk cartons to provide two composite samples per plot. For determination of primary or waterstable aggregates by the wet sieving technique, the 1 – 2 mm (or 'C') fraction of the dry aggregate analysis of each subsample from every plot was used.

All hydraulically cored soil samples for physical and chemical analyses and also the surface samples collected manually for dry aggregate analysis were air dried and stored at ambient temperature until analyzed. Surface samples collected for microbiological and hydrocarbon analyses were always stored field moist at 0°C.

4.4.2 Collection of Plant Samples

To assess the effect of soil incorporation of oily waste sludge and subsequent biodegradation or bioconversion as well as oil-related fertilization on plant growth, cereal crop plants were counted and sampled at various stages.

2. When the durum had reached anthesis (full bloom) in 1998 and 1999 a representative rectangular area of 1.0 m2 (0.5 m wide x 2.0 m long) was selected for sampling in each plot seeded to durum and oats. The plants were hand-cut, counted and bagged. The durum and oat plants were cut at anthesis to measure vegetative dry matter production by the cereal crops. After each collection of green plants and transportation to SPARC, the material was dried in a forced air oven at 40°C for at least 72 h and weighed. The dried plants were then ground and submitted to the chemistry laboratory for analysis for total N and P.
3. When the oats and durum reached grain maturity in 1998 and 1999, the plants of a one metre length within a row were hand-cut, counted and bagged. After transportation to SPARC, the plant sample was dried, weighed and then threshed by hand. The resulting grain and straw was weighed from which straw DM, straw N & P content and harvest index was determined. From each plot seeded to a crop, the ends of the plot were trimmed using a Wintersteiger harvester and then a strip was

harvested down the middle of each plot. The grain from this area was used to determine grain yield, N & P content of grain, test weight and 1000 kernel weight.

In 1998 almost all the O2F treatment plots as well as a few O1F plots produced no durum or oat plants available for hand- or mechanical harvesting.

4.5 GROUNDWATER SAMPLING

The groundwater at the project site near Richmound, SK was collected on three dates from six piezometers over the course of the experiment. The water was collected using disposable bailing tubes. At each piezometer, water was bailed three times before the water sample was collected and placed in a one litre glass bottle. The bottles were then sealed with a cap. The samples were stored at 0 C in the dark, transported to Enviro-Test Laboratories in Edmonton, AB were they tested for PAHs, except the final sampling which was analyzed for nutrients (NO3-N, NH4-N, PO4-P and SO4-S) at the Analytical Chemistry Laboratory at SPARC. The locations of the piezometers at the study site are shown in Figure 2.1.

4.6 AGROMETEOROLOGICAL MEASUREMENTS

To monitor weather conditions during the growing season of the cereal crop, appropriate instruments were transported each year to the site near Richmound, installed on a central location at the site (i.e. in the roadway just east of plots 18 and 24) prior to seeding and were dismantled and returned to SPARC each fall after the final soil sampling (October).

The on-site agrometeorology data was collected electronically with a Campbell Scientific, Model 21X micrologger and a 32 channel multiplexer powered by a 12 v car battery and contained in a protective metal box with rodent-proof lead-ins for connection to several instruments. Temperature probes were placed at a 1.0 m height above the ground and at 5 cm soil depth in four locations in each of four plots (plots 18, 24, 61 & 67). Effect of soil oiling was assessed by monitoring soil temperature at 5 cm depth, i.e. at the center of the oil incorporation depth, in both oil treated and control soil. The other instruments at the site were an automatic tipping bucket rain gauge for precipitation records, an anemometer at 2.0 m height for wind speed and a pyranometer for measurement of incoming solar radiation. Some agrometeorology data from 1998 are missing because of temporary malfunction of the 21X micrologger.

5. ANALYTICAL METHODS

5.1 SOIL PHYSICAL ANALYSES

The effectiveness of erosion proofing of sandy soils as a means of disposal of oily wastes can be evaluated by measuring the magnitude and persistence of increases in the aggregation of soil particles after application and incorporation of the oily waste sludge.

5.1.1 Soil pH and Electrical Conductivity

A saturated soil-water paste was prepared with 100 g air-dry and sieved soil (<2 mm) using de-ionized water as described by Rhoades (1982). For pH measurement the electrodes were inserted into the soil paste according to McLean (1982), pH was read immediately on a standardized pH meter and recorded as soil pH in water. The paste was then transferred onto Whatman No. 42 filter paper in a Buchner funnel and filtered under vacuum into a test tube. Electrical conductivity, a means of estimating total salt concentration, was determined on the saturation extract (filtrate) in the dip-type cell of the conductivity meter and recorded as deci-Siemens per meter (dS/m).

5.1.2 Bulk Density

Soil bulk density is the ratio of the mass of dry solids to the bulk volume of the soil. It varies with structural condition of the soil and hence is often used as an indirect measure of soil structure. The mass of soil contained in the undisturbed 10 or 15 cm x 4.75 cm cores of surface soil was determined as described by Blake and Hartge (1986). The bulk density was recorded as Mg/m^3.

5.1.3 Particle-Size Analysis

Particle-size analysis provides a measurement of the size distribution of individual or ultimate particles in a soil sample. It is based on the dispersion of soil aggregates into discrete units and the separation of particles according to size limits by sieving and sedimentation. Soil particles smaller than 2.0 mm are divided into three major size groups: sands <2.0 – 0.5 mm), silts (<0.5 – 0.002 mm) and clays (<0.002 mm). Different quantitative combinations of sand, silt and clay separates in a soil are the basis of its textural classification. Since texture is one of the most stable soil characteristics and essentially unaffected by cultivation and other agronomic practices only the benchmark samples taken to a depth of 120 cm in Spring 1998 were analyzed for particle size distribution.

In preparation for particle-size analysis 100 g samples of sieved (<2.0 mm) and dried soil were placed in large beakers and soaked overnight in a 1% solution of sodium hexametaphosphate as dispersing agent. Then the soil suspensions were vigorously stirred for 12 minutes with electric mixers and after transfer to sedimentation cylinders particle-size distribution was determined by taking multiple measurements on the same suspension by the hydrometer method as described by Day (1965).

5.1.4 Aggregate Stability and Size Distribution

An aggregate is a group of primary particles that cohere to each other more strongly than to other surrounding soil particles. Disintegration of the soil mass into aggregates requires the imposition of some disruptive force (e.g. shearing forces used during soil sampling and sample preparation, abrasive forces during sieving and/or forces evolved during the entry of water into the aggregate). Stability of the aggregates is a function of whether the cohesive forces between particles can resist the applied disruptive force.

The size of soil aggregates determines their susceptibility to movement or erosion by wind and water and aggregate size also determines the dimensions of the pore space in cultivated soils. Pore size affects movement and distribution of water and air in soil which are major influences on microbial activity and plant growth.

5.1.4.1 Dry Aggregates

The mechanical stability of dry aggregates, as determined by sieving, is a good index of the susceptibility of soil to erosion by winds. Chepil and Bisal (1943) devised a rotary-sieve machine to standardize the sieving procedure for dry aggregate size distribution. Rotary dry sieving is a sensitive measure of soil structure differences due to amendment, fertilization and cropping practices (Chepil 1962, Campbell et al. 1993a).

In preparation for dry sieving, the composite surface samples (cf. 4.4.1) were removed from the two litre milk cartons, dropped from a 1.0 m height to effect the break up of large clods, and then spread out to air dry. Aggregate size distribution was determined using the rotary sieve as described by Chepil and Bisal (1943). This process separated the soil into seven size fractions, i.e., <0.42 mm (A), 0.42-0.84 mm (B), 0.84-2.0 mm (C), 2.0-6.4 mm (D), 6.4-12.7 mm (E), 12.7-38.0 mm (F), and >38 mm (G). Fractions F and G were collected together in the rotary sieve used. Each aggregate size fraction was weighed and recorded. The wind-erodible fraction was estimated by combining the weights of fractions A and B and was reported by expressing the weight of the erodible fraction (<0.84 mm) as % of total dry weight of soil.

5.1.4.2 Waterstable Aggregates

The waterstable aggregates were determined by utilizing the C fraction (0.84-2.0 mm) from the dry aggregate analyses as described by Kemper and Rosenau (1986). Four grams of the air-dried C fraction from dry aggregate analyses were weighed into a sieve cup which was then placed into a carousel that oscillates up and down in a water cup. The samples are raised above and lowered into the water for 3 min at a rate of 35 strokes per minute. The water cups, containing particles and aggregate fragments that have broken loose from aggregates and passed through the sieve, are then placed into a 105°C oven for at lest 24 h to dry. The sieve cups (containing the stable aggregates) are then placed into a second water cup and the contents are sonicated with an ultrasonic frequency probe for 30 s to disrupt all stable aggregates and allow the resulting fragments to pass through the sieve. The second water cup for each sample is then also placed into a 105°C oven for at least 24 h to dry. The contents of the two sets of water cans are weighed after drying and the two fractions used to determine % waterstable aggregates.

5.2 SOIL AND PLANT CHEMICAL ANALYSES

The following soil and plant chemical analyses were conducted by the SPARC analytical chemistry laboratory. The details of all analytical procedures are described in a methods manual used by the above laboratory (Winkleman 1998). For some analyses, the segmented AutoAnalyzer technique was employed. Sample transmittance values are compared against the relevant standard chemical calibration curve and sample concentrations are then calculated. All soil samples undergo a saturation paste extraction step.

5.2.1 Soil Organic Matter (Total C & N)

5.2.1.1 Total C

Total carbon was determined by the dry combustion method of heating the sample to 925°C in a Lindberg furnace where CO_2 free air is drawn over the sample and through a sodium hydroxide tower where the evolved CO_2 is trapped. The dissolved carbonates are precipitated out, then the residual hydroxide ions in the NaOH reagent are determined by titration against a standardized HCl solution. The decrease in the concentration of hydroxide ions in the NaOH reagent is proportional to the CO_2 evolved during sample combustion (McKeague 1978).

5.2.1.2 Total N

The sample is digested in hot concentrated sulfuric acid (400°C) containing potassium sulfate and selenium to increase the reaction temperature and to accelerate digestion. The sample is made alkaline and then distilled to a 4% boric acid solution. A potentiometric titration is conducted using standard 0.02 M HCl. The per cent N is calculated according to the following equation: % N = [14.0067 x (sample titre - blank titre) x molarity of acid]/weight of sample (g) x 10 (McKeague 1978).

5.2.2 Plant Available Nutrients

5.2.2.1 Nitrate and Nitrite - N

The procedure for the determination of nitrate plus nitrite N was the hydrazine reduction method. After reduction the product is mixed with sulfanilamide to form a diazo compound that is then chemically coupled to the sulfulanilamide reaction product to form a coloured compound. The sample is then combined with a copper sulfate catalyst to effect a further reduction of NO_3^- to NO_2^-. The nitrite is then calculated from the difference in the values after the final reduction step was employed (Hamm et al. 1970).

5.2.2.2 Ammonium - N

Ammonium - N was determined by the ammonium salicylate complex method. The extract was combined with a nitroprusside reagent and then oxidized by sodium hypochlorite (NaOCl). During oxidation two salicylate groups combine with one nitrogen atom to form an indosalicylate sodium salt (Gentry and Willis 1988).

5.2.2.3 Phosphate - P

Soil inorganic phosphorus was determined by the acidic molybdate/ascorbic acid method. The sample was combined with ammonium molybdate and L-ascorbic acid under acidic conditions. Dodecamolybdophosphoric acid is reduced by ascorbic to the heteropoly 'molybdenum blue' (Hamm et al. 1970).

5.2.2.4 Sulfate - S

The sample is passed through a Dowex 50W-8X ion-exchange resin column to remove calcium, aluminum, lead, strontium and iron. The sample is then mixed with $BaCl_2$ at a pH of 2.5 to 3.0 to precipitate $BaSO_4$. After mixing, the pH is raised to 12.5, causing the excess Ba++ ions to complex with methylthymol blue. The non-chelated methylthymol blue in solution is equivalent to the quantity of sulfate removed during the precipitation step (Hamm et al. 1970).

5.2.3 Plant N and P

5.2.3.1 Total N

Crude protein nitrogen analysis was performed using a sulfuric acid/Se/Na_2SO_4 plant tissue digest. The digest is combined with an oxidizing agent (NaOCl) and sodium salicylate to form the coloured indo salicylate sodium salt. Sample per cent transmittance values were compared against a standard ammonium sulfate calibration curve and then the sample concentration was calculated (Williams 1984, Gentry and Willis 1988).

5.2.3.2 Total P

For phosphorus analysis the digest of plant tissue was combined with ammonium molybdate under acidic conditions. The resultant complex was then reduced with ANSA to form a coloured product called heteropolymolybdenum blue (Fiske and Subbarow 1925, Varley 1966).

5.3 MICROBIAL ANALYSES

5.3.1 Plate Count of Bacteria and Actinomycetes

Populations of viable aerobic heterotrophic bacteria and of actinomycetes were determined by the dilution surface spread plate method, on pre-poured soil extract agar (SEA) medium made from Hatton loamy sand. Five plates each were inoculated at two dilution levels per sample with 0.1 ml of dilute soil suspension and spread over the agar surface with a flame-sterilized bent glass rod shaped like a hockey stick. The plates were allowed to adsorb the inoculum for approximately five minutes before they were inverted and placed in metal boxes for incubation at 21°C for 14 days in the dark. After incubation all the plates were first examined for any obvious differences in colony morphology or other population traits across treatments. The colonies on each inverted plate were then individually counted to determine the total number of colonies per plate using a New Brunswick colony counter (1.5 X magnification). The plates were then turned right side up and the lid removed to begin selective visual counting of the actinomycete colonies only. The bacteria and actinomycetes were differentiated on the basis of differences in colony characteristics as evidenced by probing with a bacteriological needle. The actinomycetes typically have a firm, brittle or powdery consistency and some can attach tenaciously to the agar. They are quite distinct from the more mucoid or slimy bacterial colonies. The total count less the actinomycete count provides the bacteria count. All counts were expressed on the basis of 1 g of oven-dry soil (Wollum 1982, Biederbeck and Jame 1984).

5.3.2 Plate Count of Filamentous Fungi and Yeast

The surface spread plate method on pre-poured selective and differential rose bengal streptomycin agar (RBSA) medium, was used to enumerate populations of viable fungi and of yeasts. Five plates each were inoculated at two dilution levels per sample with 0.1 ml which was aseptically spread over the agar surface with a bent glass rod shaped like a hockey stick. The plates were allowed to adsorb the inoculum for approximately five minutes before they were inverted and placed in metal boxes for incubation at 21°C for 6 days in the dark. After incubation all the plates were first examined for any obvious differences in colony morphology or other population traits across treatments. The colonies on each inverted plate were then individually counted to determine the total number of colonies per plate using a New Brunswick colony counter (1.5 X magnification). The plates were then turned right side up and the lid removed to begin selective visual counting of only the smooth, distinctly non-filamentous yeast colonies. Some colonies had to be probed with a bacteriological needle to confirm their structural characteristics. The rather compact yeast colonies typically appear as light rose to reddish in colour and have a soft buttery consistency in contrast to the much larger colonies formed by the rapid growth and fluffy appearance of the mycelia of filamentous fungi. The total count less the yeast count provides the filamentous fungi count. All counts were expressed on the basis of 1 g of oven-dry soil (Wollum 1982, Biederbeck and Jame 1984).

5.3.3 Plate Count of Oil Utilizing Microorganisms

A selective and differential agar spread plating method to determine the number of viable oil utilizing microorganisms was developed. Oil agar was made using the protocol modified from Atlas (1995). Oil powder was prepared by adding 10 g of the hydrocarbon source (i.e. refinery oily waste sludge) to 30 ml of diethyl ether. This mixture was then added to 10 g of silica gel (100 to 200 mesh) and the ether was allowed to volatilize in a fume hood leaving behind a tarry, granular "powder". For each litre of medium 10 g of oil powder was added to 500 ml of distilled water, heated and stirred until well dispersed. Another 490 ml of distilled water was then added followed by 20.0 g agar, 3.0 g NaCl, 1.0 g NH_4NO_3, 0.5 g $MgSO_4$, and 0.1 g $FeCl_3$. Solutions containing 10% K_2HPO_4 and KH_2PO_4 were prepared in separate containers. The mixtures were autoclaved for 15 min at 121°C (15 psi) and then cooled to 45-50°C. Additions of 7.0 ml of K_2HPO_4 and 3.0 ml of KH_2PO_4 solutions were then made aseptically. The resulting agar had an opaque, grey background colour with dark brown to black granules uniformly distributed throughout. For this analysis, only prokaryotes (bacteria and actinomycetes) were examined using soil dilutions and inoculation techniques analogous to those of the general plate count for bacteria and actinomycetes. Inoculation and incubation was the same as for the SEA plates (cf. Section 5.3.1), however only counts of total procaryote colonies were recorded.

5.3.4 Evaluation of microbial load in CCRL wastewater sludge

The oily waste sludge was sampled on November 5, 1997 prior to, during and after application and soil incorporation at the field site near Richmound, SK. These samples were then transported to SPARC and stored at 4°C until the time of analysis. The sample taken during the application/incorporation of the oily waste was not used for this microbial evaluation since it had been already used in prior analyses and was stored at room temperature at various times. However, the remaining samples taken at pre-application and post-application were sub-sampled in duplicate. These subsamples were serially diluted and then spreadplated onto soil extract agar (SEA) for enumeration of total bacteria and actinomycetes, onto rose bengal-streptomycin agar (RBSA) for enumeration of filamentous fungi and yeast and onto oil agar for evaluation of procaryotic oil-degraders. The plates were inverted and incubated at 21°C. RBSA plates were evaluated after 6 and 14 days and SEA and oil agar plates after 14 days.

5.3.5 Soil Microbial Respiration (C-mineralization)

Soil microbial respiration was measured by the biometer flask method of Bartha and Pramer (1965) using the field moisture equivalent of 50 g oven dry weight soil per flask. The soil samples were wetted to their field capacity (equivalent to –0.03 Mpa potential) and pre-incubated, unsealed but covered, for three days to condition the soil. After pre-incubation the soils were re-wetted to field capacity if necessary. Then the large 250 ml flask was sealed with an ascarite-filtered stopcock and the 50 ml fused sidearm tube was closed by a stopper pierced with a hypodermic needle. The only modification to the procedure involved the alkali (CO_2 absorbent) exchange and recharge system. The standard 16 gauge needle was replaced by the larger 10 gauge needle to facilitate more rapid and complete, vacuum-assisted alkali collection and recharge using a repeater pipette. The alkali was exchanged after 4, 9, 16, 23 and 30 days of incubation at 21°C in the dark. The excess carbonate, formed in the alkali solution, was precipitated out by the addition of 1.0 ml of a 1M barium chloride solution and then the alkali was titrated against a standardized concentration of hydrochloric acid. The amount of CO_2-C evolved was calculated from this titrimetric data.

6. RESULTS AND DISCUSSION

6.1 WEATHER CONDITIONS AND SOIL TEMPERATURES

Weather conditions varied extensively over the 22-month period following application and incorporation of the CCRL oily waste sludge into the Hatton loamy sand. Air and soil temperatures, precipitation and wind were recorded over the growing period with the meteorological site functional from mid- April to at least late September in both years. The long-term weather data used for comparison with the site's weather data was taken from the records for Leader, a town located approximately 50 km northeast of the site. The Leader long-term weather data was used due to the incomplete weather record available for Golden Prairie, which is located only 25 km south of the site.

Mean daily air temperatures were generally similar to the long-term regional means, however in 1998 they tended to be higher while in 1999 they were slightly lower until the end of July (Table 6.1-1).

Total precipitation was quite similar to the regional long-term mean, however in 1998 all months had very low precipitation except June. The high precipitation in June is attributed to one single rain event over a 26 h period on June 26-27, which resulted in over 80 mm of rain. The very sandy soil was not able to retain much of the moisture from this rain event and the 1998 field season was under drought stress for most of that period. These drought conditions resulted in very poor germination and crop growth in 1998. The site had to be reseeded in late May 1998 due to very poor emergence of the durum and oats, however, the continuing drought still led to very poor growth. August and September were the two months with the greatest precipitation shortfall compared to the long-term regional mean (Table 6.1-1).

Temperatures within the soil tend to be less variable than air temperatures yet they are important because they influence rates of biological and chemical processes and

Table 6.1-1. Weather data for growing season of 2 yr period (1998 and 1999) at field site near Richmound, SK and long-term means from an AES climate station at Leader, SK.

Year	May	June	July	Aug.	Sept.	
	Mean Daily Air Temperature, °C					
1998	13.7	14.5	19.9	21.9	19.1*	
1999	10.3	14.9	17.4	19.6	11.7	
40 yr mean at Leader	12.1	16.7	18.9	18.1	11.8	
	Monthly Precipitation, mm					
						Total
1998	16.3	124.2	33.3	14.7	4.8	193.3
1999	71.4	69.6	56.4	20.1	1.8	219.3
40 yr mean at Leader	38.7	57.8	50.8	40.6	31.4	219.3

* Data collected in 1998 only until September 18th.

the rate of crop growth. The absorption or reflection of solar radiation during daytime by surface soil is determined primarily by surface vegetation, soil colour and moisture. Land application of oily wastes will modify temperatures at ground level and in the surface soils by darkening the soil colour and reducing or eliminating surface vegetation resulting in decreased reflectance and increased absorption of solar energy during the daytime (Rowell et al. 1992). The effect of soil oiling was assessed by monitoring soil temperature at 5 cm depth, i.e. the center of the oil incorporation depth, in both oil treated and control soil. In the first field season after oil incorporation, soil temperatures tended to be quite similar, however, the oiled soils were usually slightly higher in temperature (Figure 6.1-1). Most of the time the differences between the oiled and control soils were less than a degree throughout the growing season. These temperature differences between oiled and control soils were lower than those found in other studies (Rowell et al. 1992, Biederbeck et al. 1993). One reason for the relatively small temperature differences between oiled and control soils at this site may be due to the incomplete incorporation of oily waste (cf. Section 6.2.2) The soil temperatures were higher than the air temperature for the majority of the field season.

In the second field season, the differences between oiled and unoiled soil were quite similar to the previous year with differences of less than one degree being again the norm (Figure 6.1-2). The air temperatures were again lower than soil temperatures for most of the 1999 field season. The slight increases in soil temperature due to oil application may be beneficial to soil microbial activities and may boost hydrocarbon decomposition rates (Sexstone and Atlas 1977) or bioconversion to stable soil organic matter.

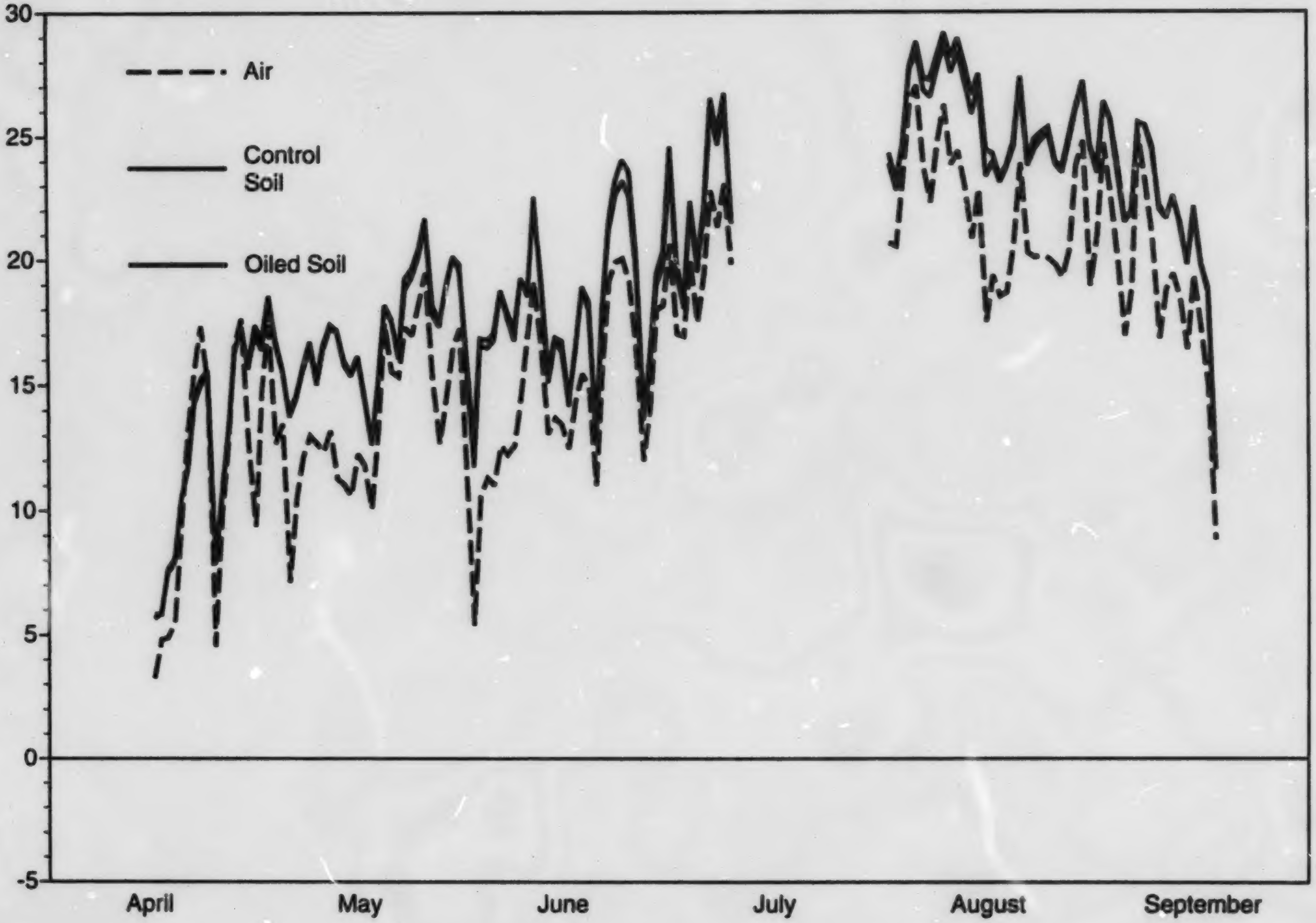


Figure 6.1-1 Daily mean air and soil temperatures (5 cm depth) at project site during 1998 growing season.

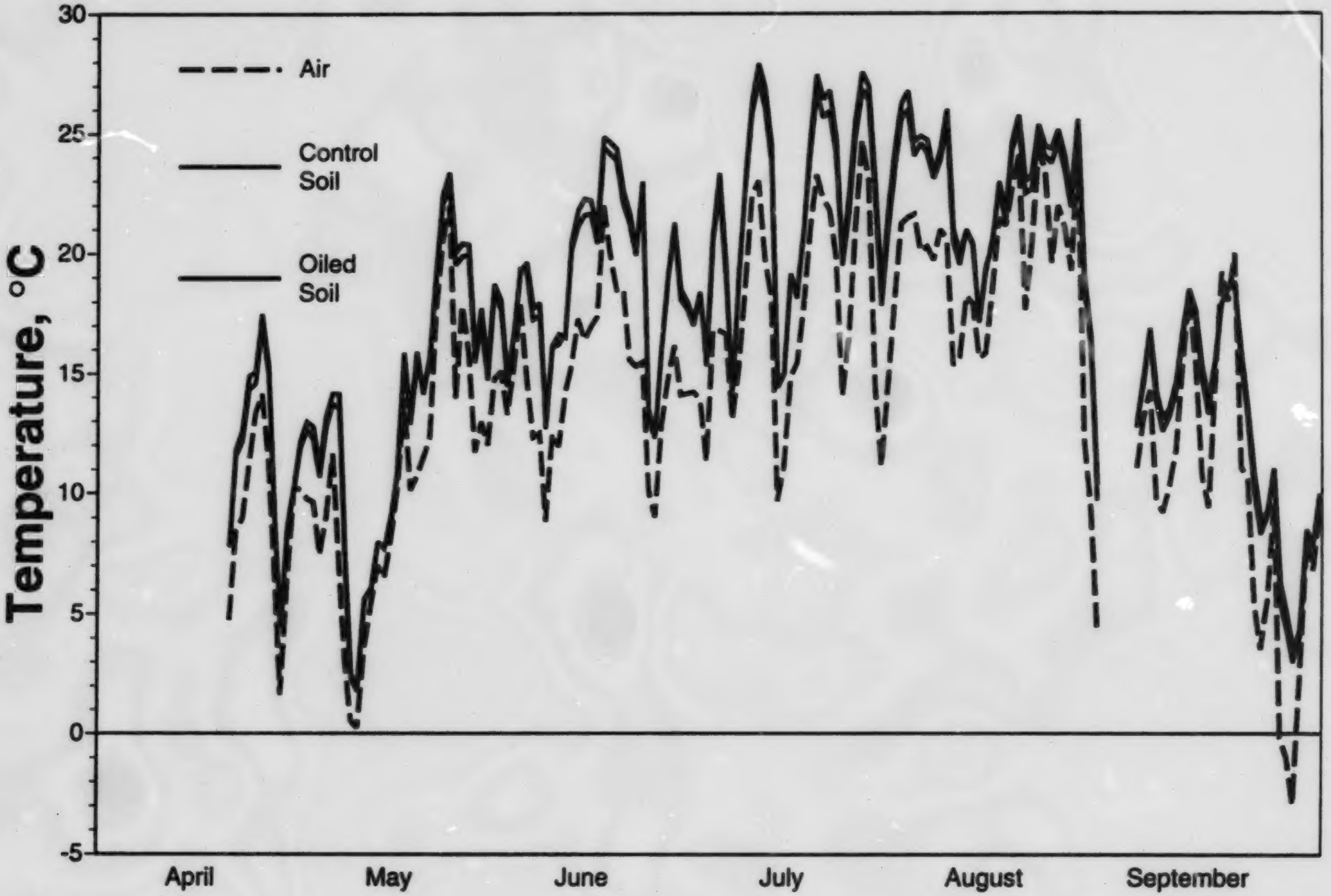


Figure 6.1-2 Daily mean air and soil temperatures (5 cm depth) at project site during 1999 growing season.

6.2 OILY WASTE CHARACTERISTICS AND DEGRADATION IN SOIL

6.2.1 Chemical Composition of Oily Waste Sludge

The oily waste sludge utilized in this study came all in one tanker truckload from storage tanks at the Consumers' Co-operative Refineries Limited (CCRL) in Regina, SK. Since the material was only from one tanker truckload and was transferred by pumping from the tanker to a vacuum truck, thus mixing the material, the amount of HC being added to the separate field plots is believed to be fairly consistent across the study area. From the analysis provided, approximately one-third of the waste water sludge was HC (Table 6.2-1).

Table 6.2-1. Gross composition of waste water sludge hauled in 1997 from Consumers' Co-operative Refineries Limited for application/incorporation in the field experiment near Richmound, SK.

Component	Weight %*
Water	61
Hydrocarbons	31
Solids (clay)	8

* Based on analysis of samples using the Dean and Stark method at the Enviro-Test Laboratories in Saskatoon.

The suitability of oily waste sludges for use as a soil amendment on marginal cropland depends not only on the nature of the oily materials but also on potential hazards from non-hydrocarbon constituents present in the wastes. These risk factors include high alkalinity, acidity and soluble salts (Rowell et al. 1992). The pH of this sludge was 6.6 and thus relatively neutral. The CCRL oily waste sludge was practically salt-free with only 280 ppm of sodium and chloride (Table 6.2-2). The very low salt content made this oily waste very suitable for application to sandy cropland as was done in this study.

The use of carbon-rich oily sludges on farm land as a biological treatment and disposal alternative raises environmental concerns primarily due to the nature and fate of aromatic oil components that may be toxic, carcinogenic or mutagenic such as some polynuclear aromatic hydrocarbons (PAH), phenols and heterocyclic compounds. Generally, the rates of biodegradation in soil are faster the fewer rings are present in the chemical structure of aromatic oily waste components (Cansfield and Racz 1978, PACE 1988). The aromatic fraction in the CCRL wastewater sludge, used in our field experiment, contained a wide variety of compounds (Table 6.2-3). Although the total PAH concentration was relatively high, most of these aromatic components were quite water insoluble and thus rather immobile.

Table 6.2-2. Content of other elements present in the CCRL oily waste sludge applied to the field site near Richmound, SK.

Salts	Concentration*
Sodium	280 mg/L
Chloride	280 mg/kg
Nutrients	
Nitrogen	70 mg/kg
Phosphorus	20 mg/kg
Sulfur	6000 mg/kg

Based on analysis at the Enviro-Test Laboratories, Saskatoon.

Table 6.2-3. Identified constituents of the aromatic fraction in oils from the CCRL wastewater sludge applied in the field study near Richmound, SK.

Compound	Concentration, µg/g dry wt. of sludge*
Dimethyl naphthalenes	1500
Methyl naphthalenes	1300
Benzoanthracene/Chrysene	730
Naphthalene	410
Phenanthrene/Anthracene	380
Benzopyrene	340
Methyl anthracenes	340
Pyrene	250
Benzoperylene	240
Benzofluoranthene	160
Dibenzoanthracene	140
Fluorene	100
Indeno (1,2,3-cd)pyrene	64
Fluoranthene	55
Carbazole	54
Acenaphthene	31
3- Methylcholanthrene	29
Dibenzofuran	18
Acenaphthylene	<5
Pentachlorophenol	<5
Total	~6150

* Based on analysis of samples at the Enviro-Test Laboratories in Saskatoon.

It is also possible that due to the polycyclic structure of many of these compounds, they may become readily incorporated into soil humic compounds which are known to have similar structures within the stable aromatic core of humic acids (McGill et al. 1981) and thus the PAHs may become biologically inactivated. This sludge was relatively low in volatiles as it contained only 420 ppm in total.

The wastewater sludge was also analyzed for metallic constituents, however, the vast majority were below detection levels of 0.2 mg/L with the highest metal present being nickel at 2.16 mg/L (data not shown).

6.2.2 Soil Incorporation and Degradation of Oily Waste Sludge

Due to the relatively high oil content in the waste water sludge and the low soil oiling target rates (1 and 2%), the distribution of oil within the soil layer of incorporation was not completely uniform. The target oily waste loading rates were 3.96 L/m2 and 7.92 L/m2 (the latter rate required two consecutive runs with the applicator across the plot). These small application volumes required the applicator/rotovator to be run at low speed (approximately 200 rpm) while the tractor that pulled the rotovator traveled relatively quickly (~4.3 km/h). This combination resulted in visible oil clumps having formed on the surface of the plots especially in the 1% target soil oil content plots. The 2% oil plots that required a second pass with the rotovator appeared to have more uniform oil incorporation. Repeated measurements of the time the applicator/rotovator was in each plot revealed that the actual oil loading rates were slightly less than targeted (Table 6.2-4). For ease of treatment identification the targeted initial soil oil contents (i.e. 1% and 2%) were always used when discussing these treatments.

Incorporation of the sludge changed the appearance of the Hatton loamy sand particularly in the twice rotovated plots (2% initial soil oil content or O2 treatment) that had more uniform oil incorporation and greater darkening in colour due to the oily waste addition. Immediately after incorporation, the odor of the sludge was very strong, however' it dissipated overnight, due to the low total volatile content in the oily waste, allowing for safe surface soil sampling in the plots the next day.

Table 6.2-4. Theoretical initial oil contents and the actual amounts applied as CCRL waste water sludge on November 5, 1997 to the top 10 cm of soil at the field site near Richmound, SK.

Target soil oil content (w/w)	Theoretical oil loading† (kg/m^2)	Actual oil loading (kg/m^2)	Actual initial oil content (w/w)
1%	1.35‡	1.24 – 1.34	0.92 – 0.99%
2%	2.70	2.48 – 2.68	1.84 – 1.98%

† Total oil loading based on waste sludge volume x 31% total oil x 1.1 specific gravity.
‡ Based on an average bulk density of 1.35 g/cm^3 in the Hatton loamy sand at 0-10 cm depth.

Oil degradation in the loamy sand was monitored by periodic soil sampling over the course of the study, however, the results of hydrocarbon extractions and the direct measurements are still pending. The level of total organic C in surface soil was monitored over the course of the study. This gives a good indication of the amount of oil C which has been removed from the soil by microbial C-mineralization (i.e. CO_2 evolution). The general trends for organic carbon content in the treated layer of soil show reductions from initially higher levels after oil incorporation (Table 6.2-5). Fertilization did not appear to affect the amount of organic-C maintained in the soil over time. The addition of the oily waste sludge to the Hatton loamy sand also seemed to exert the C saving or sparing effect on the indigenous soil organic matter that was found earlier by Biederbeck et al. (1993) after EOR production waste sludge was incorporated into a Meota loamy sand near Maidstone, SK. In spring 1998 (i.e. 5.3 months after oil incorporation) there were between 35 – 77% greater amounts of organic C in the soil however by fall 1999 (22 months after oil incorporation) this difference in organic matter content versus the control soil had increased to 44 – 111% (Table 6.2-5).

Table 6.2-5. Organic carbon content in the top 10 cm of a Hatton loamy sand at various periods after the incorporation of CCRL wastewater sludge.

Sampling Time †	Treatment O0, U	O1, U	O1, F	O2, U	O2, F
	----------	----------	%	----------	----------
Time zero	0.49	0.87	0.84	1.14	1.20
3 weeks	0.54	nd	0.93	nd	1.43
Spring 1998	0.60	0.81	0.95	1.06	1.03
Fall 1998	0.48	0.67	0.68	0.87	0.90
Spring 1999	0.46	0.67	0.68	0.83	0.78
Fall 1999	0.43	0.62	0.68	0.91	0.87

† Sampling times were those described earlier with time zero being 24 h after oil incorporation and 3 weeks being 20 days after oil incorporation.

6.3 SOIL AGGREGATION

Soil aggregation is a very important component of soil structure which is considered optimal when the soil is well aggregated and contains mostly large, stable aggregates of 1 to 100 mm diameter (Oades 1984). Stable soil aggregates benefit soils by reducing soil erosion, increasing aeration, maintaining soil tilth, and controlling soil water and nutrient dynamics.

Two distinct types methods of analyzing aggregation are frequently used; those that determine size distribution of dry aggregates and those that determine waterstable aggregates. A complete discussion of the attributes of both measurements can be found elsewhere (Biederbeck et al. 1993), however, it should be noted that both techniques may be required to properly assess aggregation because waterstable aggregates may be related more to long-term management while dry aggregates may be more sensitive to recent (<1 yr) cultural treatments (Campbell et al. 1993a and 1993b).

With over 87% sand content in the 0-10 and 10-30 cm depths the Hatton loamy sand at our site was highly susceptible to erosion by wind and water. In addition to the lack of sufficient silt and clay content to encourage aggregation, the organic matter content of this eroded loamy sand was also extremely low thus further contributing to the lack of aggregation. As determined by Biederbeck et al. (1993) oily waste sludge can initially act as a glue to stick sand grains together and increase aggregation while later the increase in humified and stable organic matter can result in long-term benefits to soil aggregation. The initial benefit of the incorporation of the oily waste became evident in spring 1998 when the oiled plots showed no sign of topsoil loss to wind erosion while the unoiled control plots were littered with many stones and pebbles that became exposed as the surrounding topsoil had been swept away by strong Chinook winds (Figure 6.3-2).

6.3.1 Dry Aggregates

Rotary dry sieving for dry aggregate analysis results in seven distinct size fractions, however, Chepil (1953) reported all aggregates with diameters >0.84 mm to be definitely non-erodible by wind and thus the wind erodible fraction (WEF) is generally considered to consist of soil aggregates <0.84 mm in diameter. Our initial analyses of dry aggregates, in the spring of 1998, found that there was a decrease in the WEF of 9% where 1% oil was applied in the fall of 1997 and a 13% decrease where 2% oil was applied (Figure 6.3-1). Oil-related fertilization did not affect WEF values (data not shown), therefore the data was bulked and presented only for oil treatments. This 9%

reduction in WEF for plots with 1% oil incorporated and at least 13% reduction for 2% oil plots was maintained, while oily waste sludge underwent biodegradation, at least until our last sampling in the spring of 2000 i.e. 29 months after sludge application. The slight reduction in WEF found for the control treatment from spring 1998 to spring 2000 may be due to the gradual formation of new aggregates after the prior soil aggregation was severely disrupted upon the rotovation of all plots (including unoiled controls) at the time of oil application/incorporation (Figure 6.3-2).

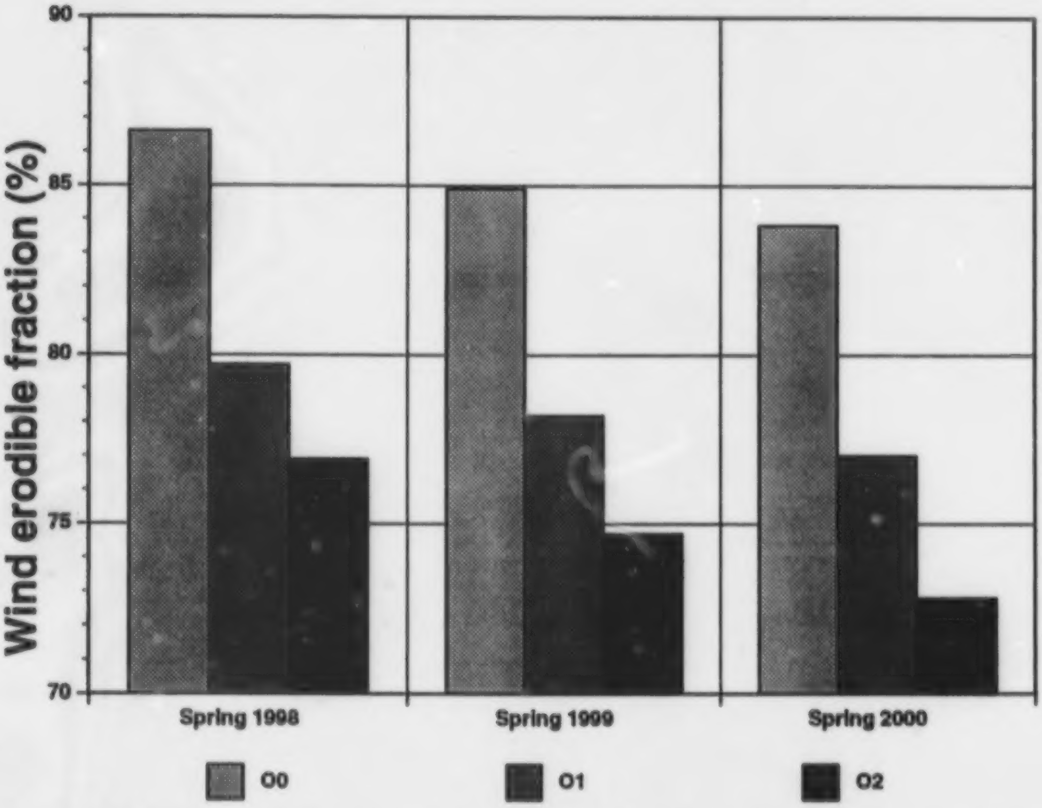


Figure 6.3-1. Effect of oily waste sludge incorporation on wind erodible fraction in the top 10 cm of Hatton loamy sand.

a)

b)

Figure 6.3-2. Appearance of soil surface a) of unoiled (at left with yellow flag) and oiled (red flag) plots and b) close-up of the unoiled plot surface in the spring of 1998 at the field site near Richmound, SK.

6.3.2 Waterstable Aggregates

The resistance to erosion by water was boosted much more than was dry aggregate stability as wet sieving analysis of the spring 1998 soil samples indicated increases of 44% and 64% in waterstable aggregates where sludge was previously applied to a 1% and 2% initial soil oil content, respectively (Figure 6.3-3). As with the dry aggregates, oil-related fertilization did not consistently affect waterstable aggregation and thus the data from fertilized and unfertilized oiled plots were bulked for analysis. The marked improvement in structure of this sandy soil was rather persistent with increases of at least 40% for 1% oil and over 66% for 2% soil oil content observed over the remaining time of the study (Figure 6.3-3). Large and persistent increases in waterstable aggregation were also found by Giddens (1976) in a field experiment with waste motor oil on a loamy sand in Georgia. Long-term benefits of oil incorporation have also been reported from some very early studies (Plice 1948) thus indicating that the breakdown products of oily materials are effective stabilizing agents for soil aggregation.

6.3.3 Aggregation Conclusions

The positive and persistent effect of incorporation of oily waste sludge on both water-stable and dry aggregates confirms that a single application can provide an effective means for stabilizing erosion-prone cropland. The immediate effects were obvious in the first spring after oily waste application/incorporation when striking visual differences were found between unoiled and oiled plots. These observed differences were backed up by the results from the analyses of surface soil samples showing significant reductions in WEF with oil treatment and large increases in waterstable aggregates. These positive effects are probably due initially to the 'binding together' of previously loose particles by

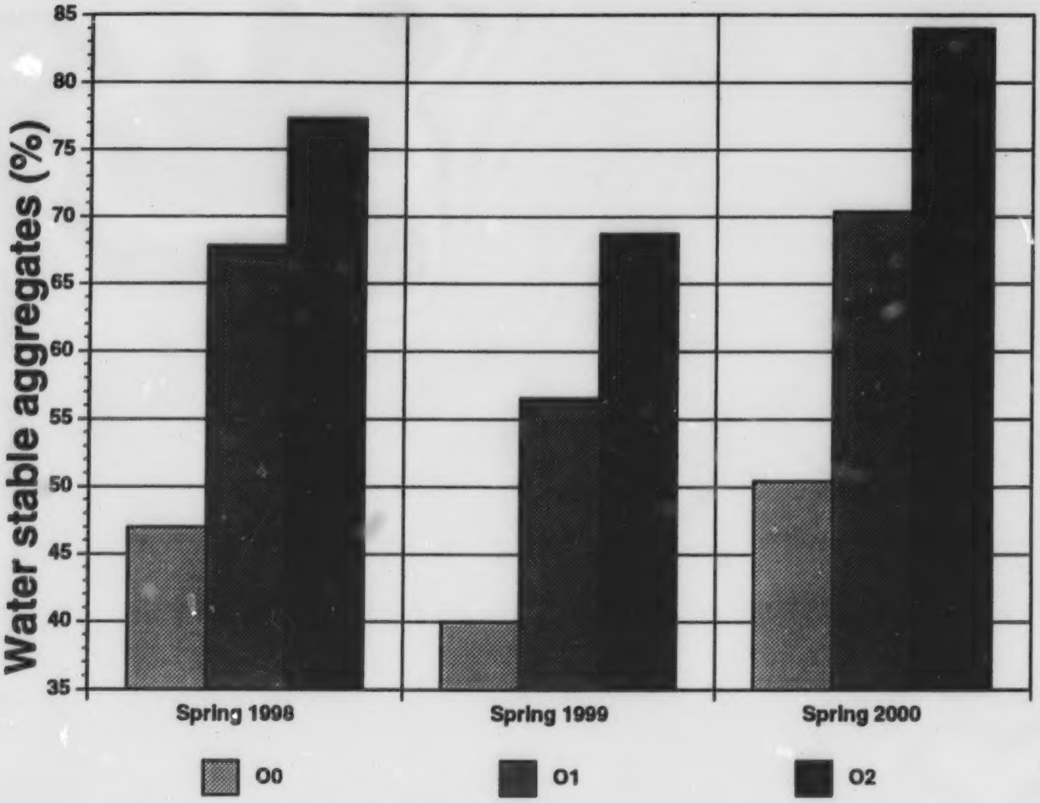


Figure 6.3-3. Effect of oily waste sludge incorporation on waterstable aggregates in the top 10 cm of Hatton loamy sand.

the sticky oil components in the sludge, as suggested by Rimmer and Al-Khafaji (1986). That said, long-term benefits as found by Biederbeck et al. (1993) are probably due to microbial bioconversion of the oily waste into microbial biomass, microbial gums, humus and other stable biodegradation products.

The long-term nature of the oil-induced aggregation benefits allows us to suggest that a single oily waste sludge application, every four to six years, should provide adequate protection against topsoil degradation by wind and water, even on very erosion-prone, light land.

6.4 SOIL MICROBIAL POPULATIONS AND ACTIVITIES

The application and incorporation of the CCRL oily waste sludge affected the soil microbial populations stimulating those deemed to be more metabolically active (i.e. bacteria and yeasts). Oil-related fertilization and the rate of oil application also influenced the extent to which the microbes were affected by the organic amendment. Microbial populations and activities are not direct measurements of HC biodegradation or bioconversion to stable humus but have been used for general evaluation of these processes (Bossert and Kosson 1997).

6.4.1 Bacteria

The numbers of aerobic, heterotrophic bacteria, recovered by plate counts, were greatly increased, even at time zero, on soils amended with the CCRL wastewater sludge (Figure 6.4-1). The initial rise in soil bacterial numbers is contrary to other reports (Riser-Roberts 1998) where microbial numbers were initially depressed after HC application. The increased numbers of soil bacteria found at time zero may be due to the processing of the soil that was required after taking the samples in the field and prior to soil dilution plating. Since the sludge becomes very viscous in cooler conditions, the samples had to be warmed to room temperature prior to rotary mixing and subsequent sieving of the soil. During this warming period it is possible that the microbial community recovered from its initial 'shock' from the hydrocarbon addition and became stimulated. Stimulation of the microbial community is found in most soils with hydrocarbon amendments (Riser-Roberts 1998). Soil bacteria responded quickly to oily waste amendment with increases of up to 300% in three weeks after incorporation. This was possible due to the warm fall weather conditions that prevailed at the site in November of 1997. Oily waste amended

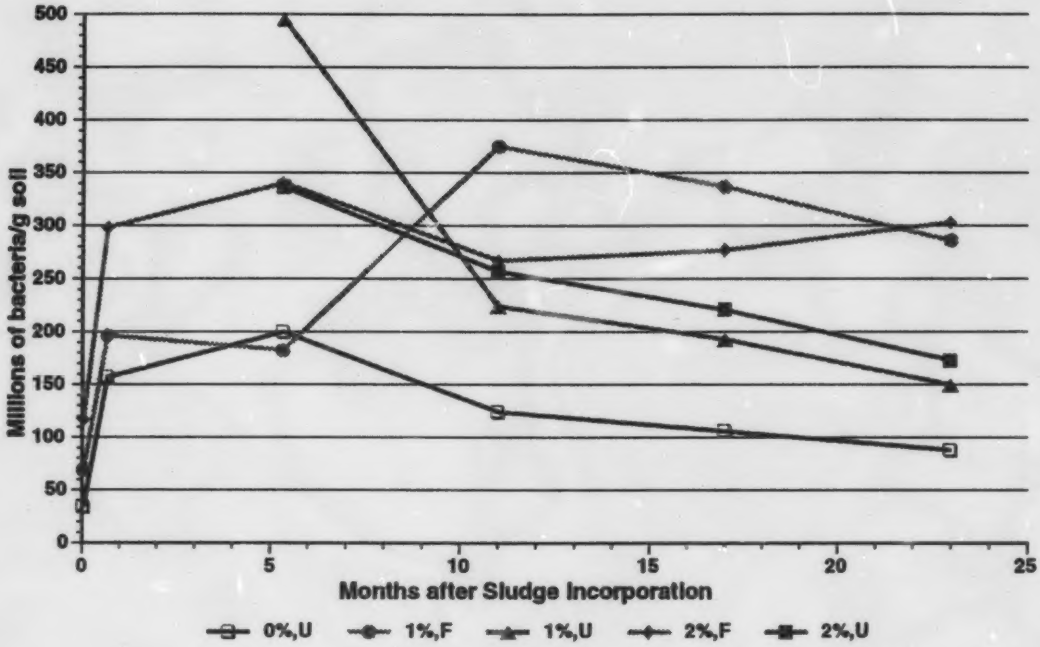


Figure 6.4-1. Response of bacterial populations over time to a single application/incorporation of an oily waste sludge in the top 10 cm of a loamy sand near Richmound, SK.

plots had consistently higher bacterial populations than did the control (0%, U) except 1%, F treatment at about 5 months after incorporation. We have no explanation for this relatively low population in the 1%, F treatment, however, thereafter bacterial numbers were increased very markedly. Bacterial populations in most treatments peaked by five months, which is sooner than was found in the earlier study at Maidstone, SK with a heavy oil production waste sludge (Biederbeck et al. 1993). At the final sampling, about 23 months after oil incorporation, bacterial populations in all oiled plots were still significantly higher than those in the control plots.

In this study, it was found that oily waste related fertilization had an important effect on bacterial populations. This was not found in the earlier PERD study, where the microbial community was not affected by fertilizer amendments (Biederbeck et al. 1993).

Bacterial populations for the unfertilized oil treatments (1%,U & 2%,U) were not determined until the spring of 1998 (~ 5 months after incorporation). For the 1% oil treatments, fertilization resulted in lower bacterial counts at 5 months but then the counts were consistently and significantly higher for the remaining period. For 2% oil, fertilization had little affect at the 5 month and 11 month sampling, however by 23 months 2%,F had significantly higher bacterial populations than 2%,U. At 23 months after sludge incorporation it appeared that three distinct groups had emerged from the bacterial population data (Figure 6.4-1): unoiled control soil with the lowest population, oiled and fertilized treatments with definitely the highest populations, and oiled but unfertilized soils with intermediate level populations. This response to N and P fertilization was consistent with field studies conducted in northern Canada (Jobson et al. 1973, Westlake et al. 1978) and incubation studies in the USA (Loynachan 1978, Song and Bartha 1990) although in these studies the response to fertilization was more quickly observed. The slow response of this trial to show significant increases in bacterial populations due to the fertilization may well be a result of the severe and persistent drought conditions during the first field season. This lack of fertilization response due to drought would be consistent with the findings in the field trial near Maidstone, SK (Biederbeck et al. 1993).

The effect of cropping on microbial populations was also examined in this field study. During the 1998 field season the drought conditions lead to very poor crop establishment which in turn would not be conducive to obtaining clear microbial responses if they existed. However, the bacterial populations present in the fallow plots in fall 1998 were significantly higher than those in the cropped plots (data not shown). This may be due to less moisture being available to the bacteria as they were competing with the plants for the little water that was available to both. After the 1999 field season, when moisture was not as limiting, cropping was found to generally effect a marked positive response by the bacterial population (Figure 6.4-2). Cropping to either durum or oats, consistently supported higher populations of bacteria in 1999 than did fallow except for the 2% oil, unfertilized treatment that was cropped to durum. In the plots cropped to durum, the bacterial response was the greatest to 1% oil when fertilization was taken into consideration while cropping to oats showed slightly higher levels of bacterial response at the 2% oil with fertilizer addition.

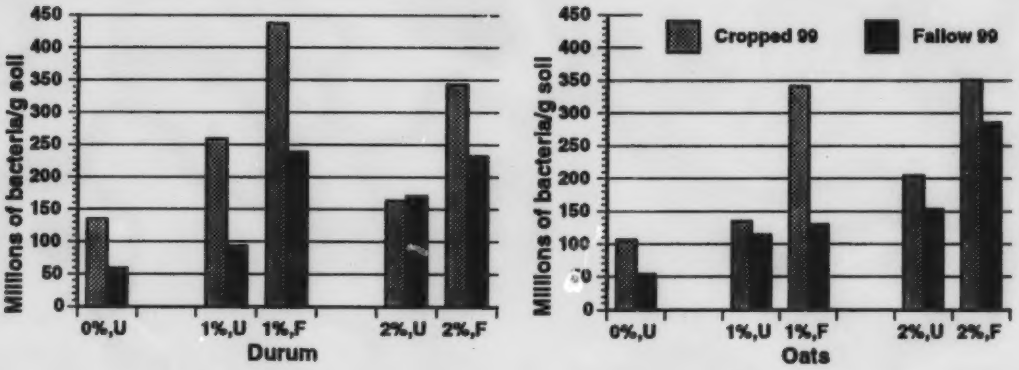


Figure 6.4-2. Response of bacterial populations in Fall of 1999 to cropping and fertilization after a single application/incorporation of an oily waste sludge in the top 10 cm of a loamy sand near Richmound, SK.

Taken together, the oil-related fertilization and cropping synergistically increased the overall bacterial populations. Fertilization had been reported to encourage microbial biodegradation of HC (with increasing microbial populations) thus speeding up reclamation and allowing cereal crop production to reach levels similar to those of the unoiled control within a year (Toogood et al. 1977). At our field site, the increased microbial populations probably did increase biodegradation and bioconversion thus allowing better crop production in the second field season which in turn again boosted the bacterial population through greater root biomass and more root exudates.

In addition to enumerating total heterotrophic bacteria, counts of hydrocarbon-utilizing bacteria (HUB) were also conducted. When the HUB were expressed as a percentage of the total bacterial population an interesting response pattern becomes evident (Figure 6.4-3). After an initially sharp rise and after about five months in the field, the HUB percentage steadily drops and at 23 months it is very close to the same level as in the control plots. Microorganisms that degrade hydrocarbons are found in all soil environments and, according to the literature, they usually "constitute less than 1% of the total microbial communities" however, when oil is present the hydrocarbon-degrading populations can increase to 10% (Atlas 1995) or even higher (Pinholt et al. 1979). At Richmound, the percentage of oil-degrading procaryotes has dropped since peaking at 44% at the three week or 33% at the spring 1998 sampling with all treatments being down to between 3 and 7% at 23 months after oil incorporation (Figure 6.4-3). This indicates that the vast majority of bacteria found in the general bacterial population do not utilize the oil directly as a C-source but may use intermediates from the hydrocarbon degradation. Thus over time, fewer and fewer HUB were required to initiate degradation of the residual oily waste and yet the overall bacterial populations remained considerably higher than the control. Thus the oil degraders in each treatment, although decreasing over time, provided enough products of metabolism to sustain significantly higher populations of bacteria on the whole.

6.4.2 Actinomycetes

The response of actinomycete populations to the single application/incorporation of oily waste sludge was somewhat similar to that of bacterial populations (Figure 6.4-5). However, the differences between the treated plots and controls were not as evident throughout the 23 months of observations. The response by the actinomycete population does not appear to be related to initial soil oil content as the population went up and down

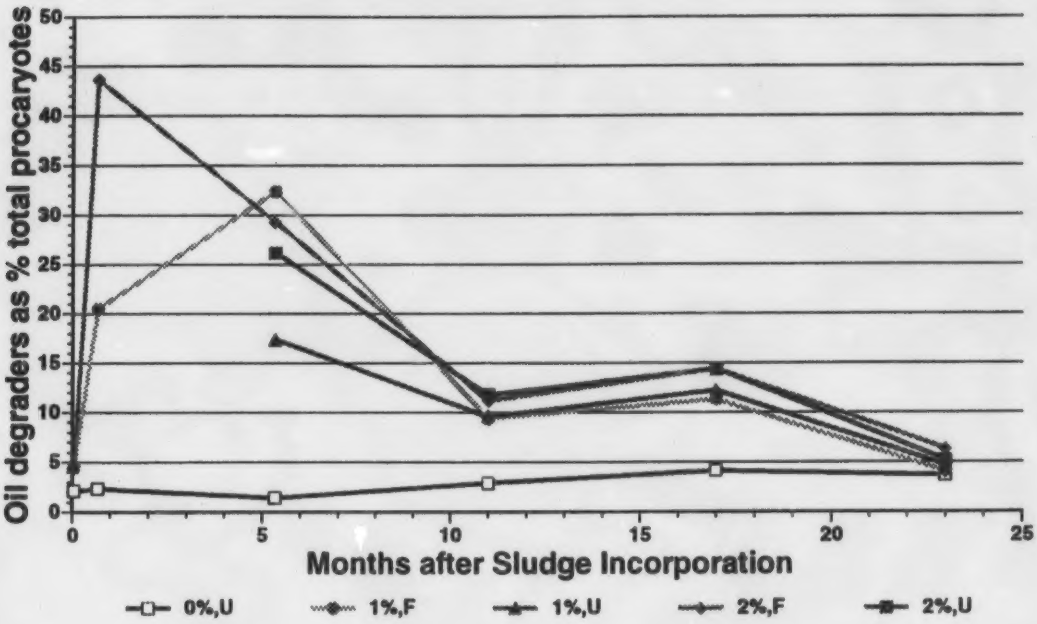


Figure 6.4-3. Response of oil degrading bacterial populations as a percentage of total bacterial population over time to a single application/incorporation of an oily waste sludge in the top 10 cm of a loamy sand near Richmound, SK.

over the course of the field study although the oiled and fertilized treatments tended to be among the highest at the 11-month mid-study point. The generally increased actinomycete populations after 11 months of the study may be consistent with the suggestion that these "autochthonous-type" organisms assume a greater role in the later stages of oily waste biodegradation in soil, when competition from "zymogenous-type" bacteria has greatly decreased (Bossert and Bartha 1984, Song and Bartha 1990). Evidence for this type of population shift in HUB has not been found in some studies (Skujins and McDonald 1985, Biederbeck et al. 1993). Actinomycete populations responded similarly to fertilization but not as consistently as bacteria and often to a lesser extent.

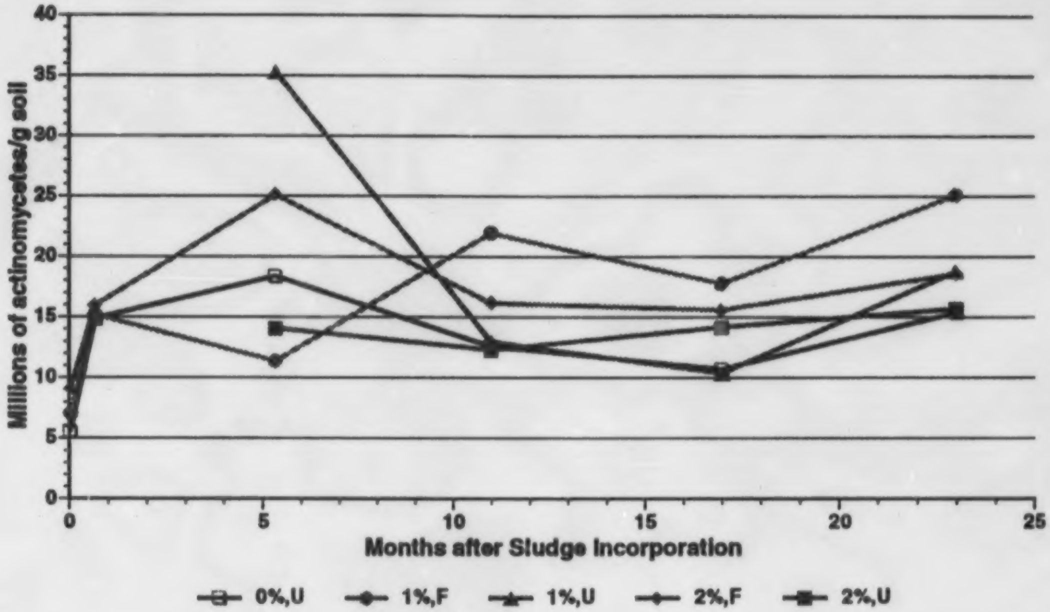


Figure 6.4-5. Response of actinomycete populations over time to a single application/incorporation of an oily waste sludge in the top 10 cm of a loamy sand near Richmound, SK.

Cropping in 1999 generally produced increased in actinomycetes compared to fallow and durum roots effected a distinct stimulation of the actinomycete population in comparison with the populations under oats (Figure 6.4-6). Actinomycete populations responded similarly to fertilization but not as consistently as bacteria and often to a lesser extent.

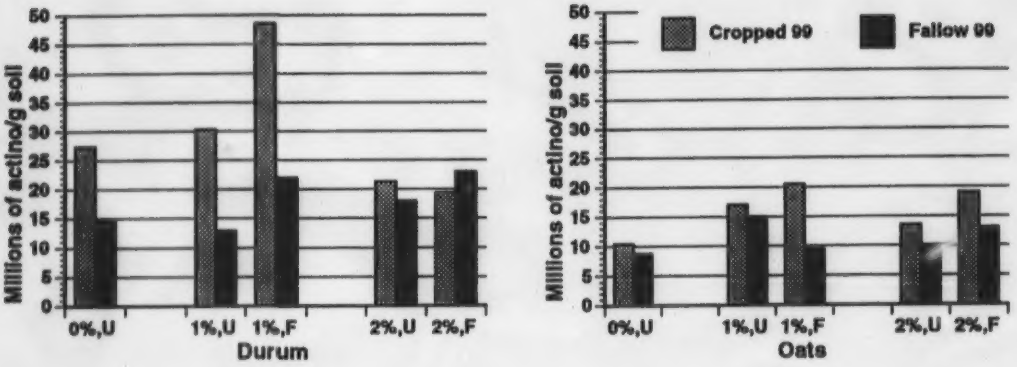


Figure 6.4-6. Response of actinomycete populations in Fall of 1999 to cropping and fertilization after a single application/incorporation of an oily waste sludge in the top 10 cm of a loamy sand near Richmound, SK.

6.4.3 Fungi

6.4.3.1 Filamentous Fungi

The effects of oil and fertilization on filamentous fungal populations were less pronounced than for the bacteria (Figure 6.2-7). The number of filamentous fungi recovered was initially increased by oily waste. Oil with fertilizer produced significantly different numbers of fungi recovered, with 2%, F consistently lower than 1%, F after the three-week sampling. Furthermore, the combination of 2% oil with fertilizer seems to be detrimental to filamentous fungi as the resultant populations were slightly below those of the unoiled control (Figure 6.2-7). Fertilization appeared to decrease the stimulation of oil application in the early portion of the study period however, 1%F treatments soon after resulted in the largest fungal populations.

General increases in fungal populations from this study are consistent with a large number of reports in the literature although these increases don't appear to be as persistent (Jensen 1975, Sexstone and Atlas 1977, Bossert and Bartha 1984, Song and Bartha 1990).

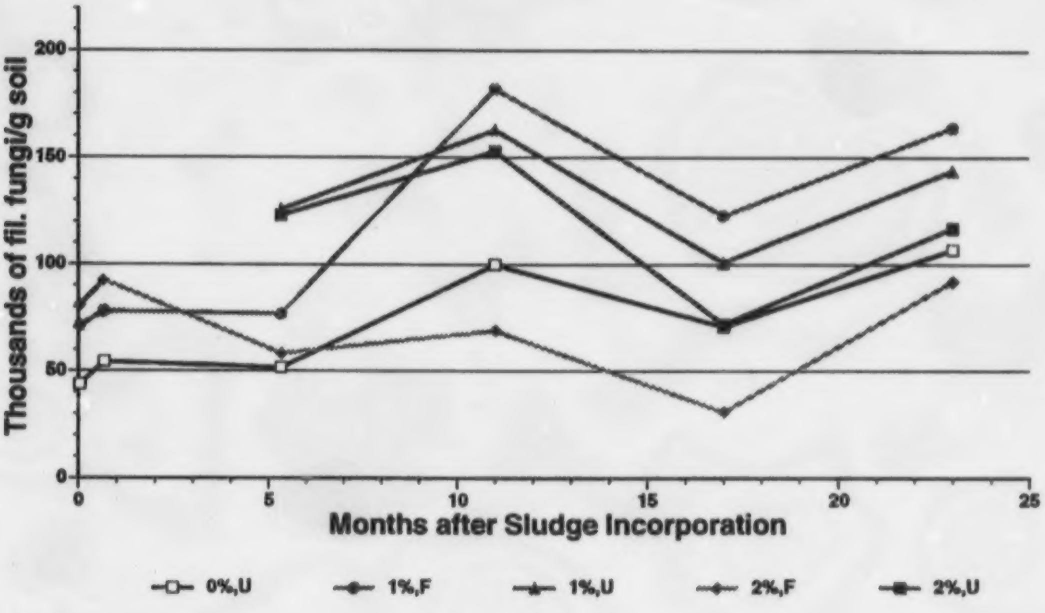


Figure 6.4-7. Response of filamentous fungi populations to a single application/incorporation of an oily waste sludge in the top 10 cm of a loamy sand near Richmound, SK.

Filamentous fungi showed little response to cropping but were affected by crop type and durum, especially at the 1% oil level, boosted the population when compared to oats (Figure 6.2-8). Fertilization responses were inconsistent among treatments for filamentous fungi (Figures 6.2-8).

6.4.3.2 Yeast

Yeast populations were delayed in their response to the oily waste and fertilization compared to other microbial populations (Figure 6.2-9). The number of yeast recovered from the oiled treatments was the same or slightly lower than that of the control until after five months from soil oiling. The yeast were slower to increase relative to the control for the 2%, F treatment compared to the other treatments and this way have been

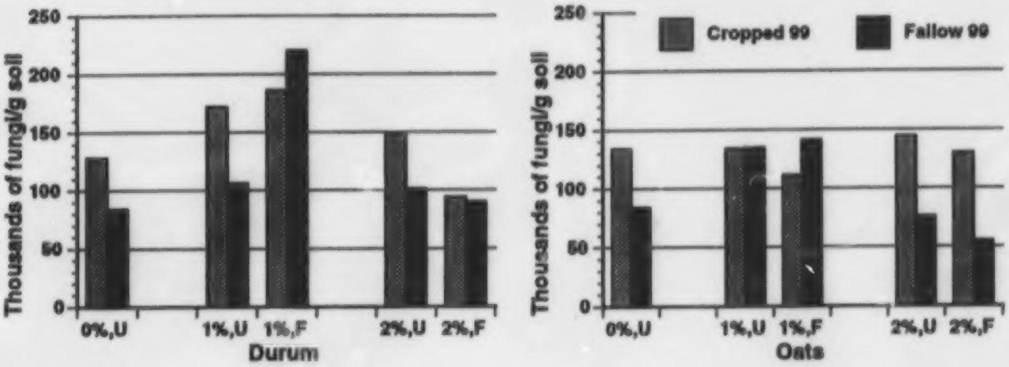


Figure 6.4-8. Response of filamentous fungal populations in Fall of 1999 to cropping and fertilization after a single application/incorporation of an oily waste sludge in the top 10 cm of a loamy sand near Richmound, SK.

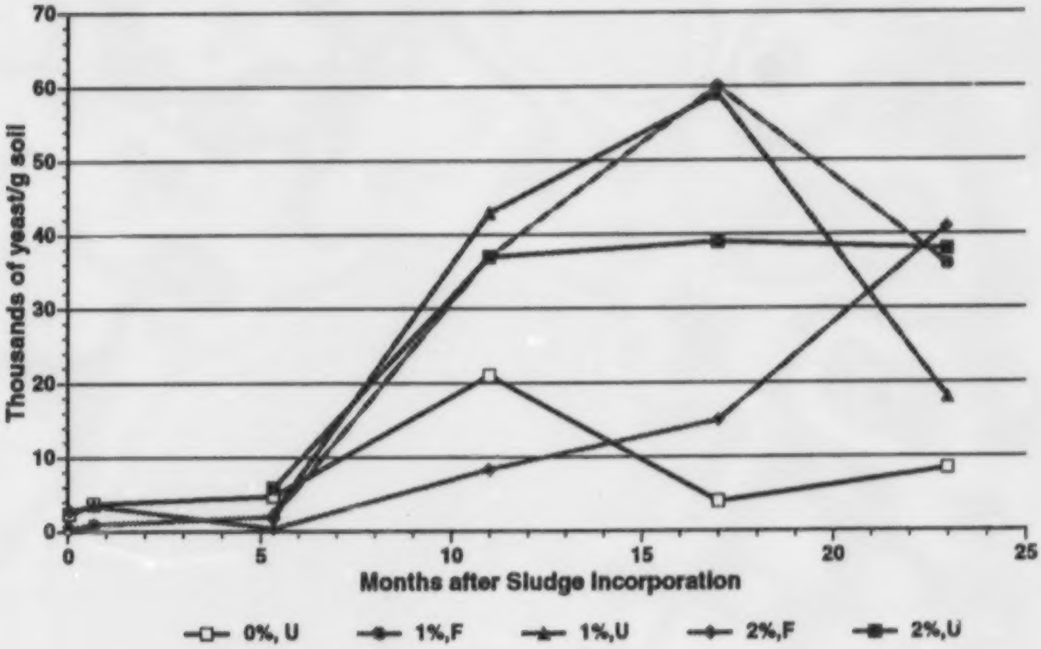


Figure 6.4-9. Response of yeast populations to a single application/incorporation of an oily waste sludge in the top 10 cm of a loamy sand near Richmound, SK.

due to the combination of relatively high oil and high fertilization which was detrimental to yeast growth. The generally stimulated yeast populations to oil application are not consistent with that found by Biederbeck et al. (1993). The stimulation may be due to the relatively low toxicity associated with the CCRL oily waste used in this study.

When the microbial population data is considered together it suggests that those microorganisms that may be characterized as more metabolically active (bacteria and yeasts) were more responsive to oil incorporation and oil-related fertilization than the actinomycetes and filamentous fungi that may often be present in resting forms like spores.

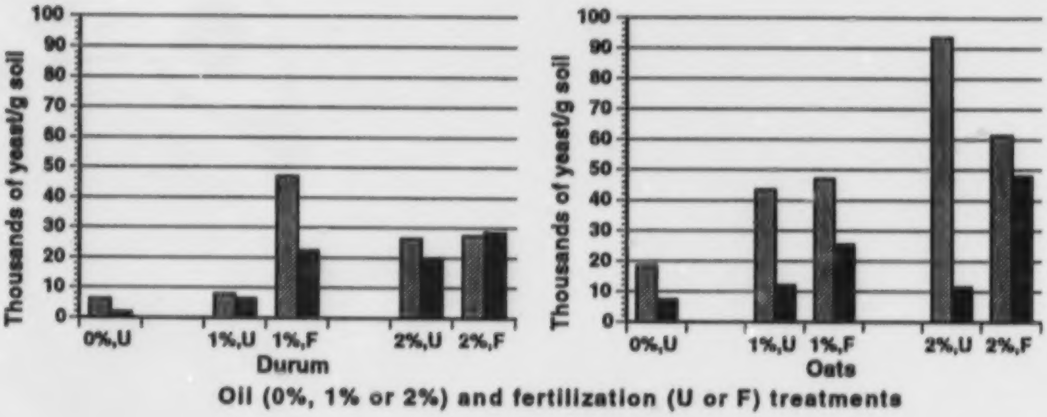


Figure 6.4-10. Response of yeast populations in Fall of 1999 to cropping and fertilization after a single application/incorporation of an oily waste sludge in the top 10 cm of a loamy sand near Richmound, SK.

6.4.4 Microbial Load in CCRL Wastewater Sludge

The fungal load in the oily waste sludge was very low with no colonies formed at the lowest dilution plated, 10^{-2} (Table 6.4-1). Since no fungi were found to have grown after six days of incubation the plates were incubated for another eight days with the same result.

The number of bacteria found in the oily waste was higher than expected with over one million per gram of oily waste sludge (Table 6.4-1). Actinomycetes were also found and those present were speculated to be *Nocardia* spp. Oil-degrading procaryotes were found to have significantly lower numbers in the post-application sample compared to the pre-application sample (Table 6.4-1).

Table 6.4-1 Microbial load in CCRL waste sludge used for soil incorporation at the field site near Richmound, SK. The samples were taken prior to and after oily waste application/incorporation on Nov. 5, 1997.

Sample	Fungi $(x\ 10^2/g)$†	Actinomycetes $(x\ 10^5/g)$	Bacteria $(x\ 10^5/g)$	Oil-degrading Procaryotes $(x\ 10^5/g)$
Pre-application	<1	0.65	39.5	29.9
Post-application	<1	0.13	13.8	5.6

† The microbial counts are expressed as per gram oily waste sludge rather than volume due to the high viscosity of the material.

Although the bacterial numbers are higher than expected, with the rate of oily waste being applied (1% or 2% (w/w)) the initial increase in bacterial oil degraders and bacteria in general can not be attributed in the oiled plots to the microbial load in the CCRL waste sludge (Figure 1). At the highest rate of 2% oily waste added, there would only be a maximum of 7.9×10^4 bacteria added to each gram soil and the bacterial counts at time zero show increases of tens of millions.

6.4.5 Soil Microbial C-mineralization

Most heterotrophic microbial activities result in production of CO_2 and thus respiration (or C-mineralization) is intimately associated with oil decomposition. Soil microbial C-mineralization was initially found to increase 2 – 2.5 fold by the addition of oil and fertilizer initially, however, after five months it was obvious that the oil-related fertilization was depressing respiration compared to treatment with oil alone (Figure 6.2-11). Similar drops in respiration rates have been reported elsewhere after addition of nitrogen fertilizer to C-rich soil (Soderstrom et al. 1983) but the reasons for these microbial responses to N fertilization are yet to be fully elucidated. After 23 months, there are no significant differences in C-mineralization rate between oil treatments with or without fertilization although the number of bacteria were shown to be about double for the oiled and fertilized treatments compared to oiled only (Figure 6.4-1). This indicates that although total respiration is the same per gram soil, on a respiration/microbial number basis the C-mineralization rate is still markedly lower for those treatments that received fertilizer. C-mineralization or respiration is often used as a measure of overall microbial activity, however, in this case it is also a measure of venting or excessive hydrocarbon degradation to CO_2 gas. With the increased awareness of greenhouse gas effects on climate, the use of adequate fertilization for more efficient bioconversion of hydrocarbons to stable soil humus (sequestered C) is now being emphasized instead of venting.

The increase in C-mineralization at 23 months after oil incorporation indicate that a great deal of heterotrophic metabolic activity is still occurring. The continued increase over this time period may be related to the drought conditions in the first 11 months of the study which depressed microbial activity overall. This elongated period of increasing C-mineralization may still be a portion o the first phase of HC degradation that utilizes the more available HC (McGill and Rowell 1977). Sampling periods or the time of incor-

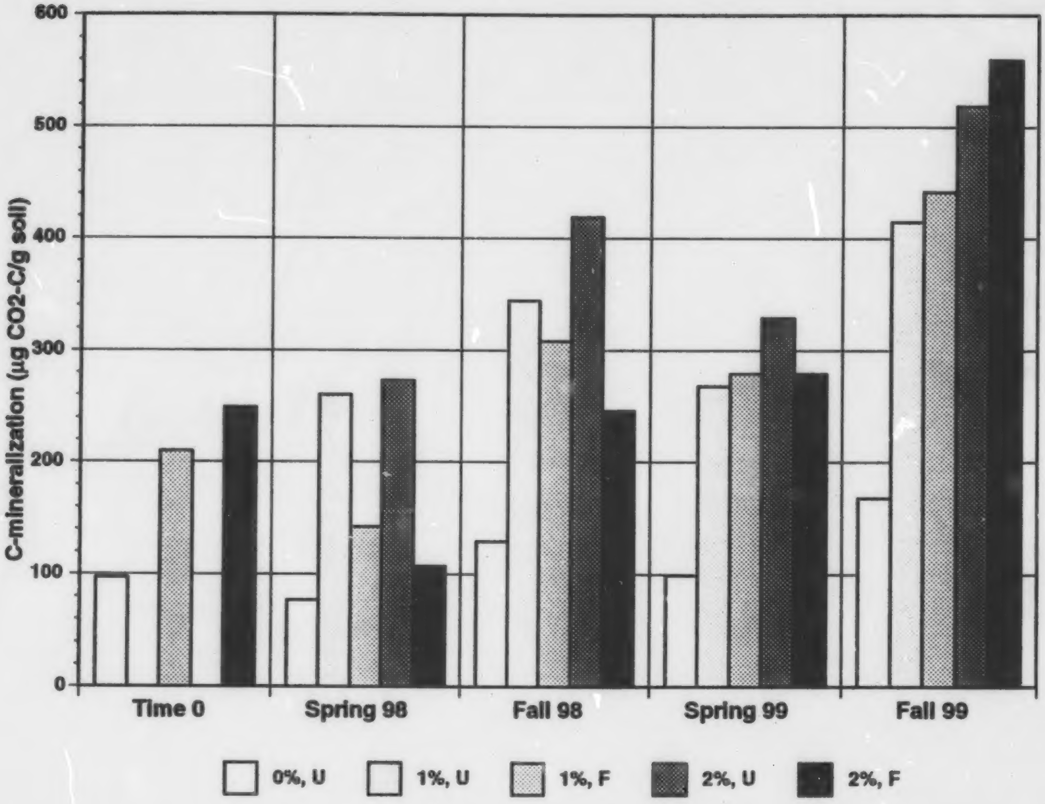


Figure 6.4-11. Response of microbial C-mineralization to the incorporation of an oily waste sludge in the top 10 cm of a loamy sand near Richmound, SK.

poration in late fall may have missed the peak of an initial flush of C-min. Other studies have found very high C-min rates in the first few months after petroleum additions to soil (Dibble and Bartha 1979, McGill et al. 1981, Bossert and Bartha 1984) even at low incubation temperatures (Loynachan 1978).

The effect of oil-related fertilization clearly shows decreases in C-min in the early stages of the study. This may be an indication of negative effects on oligotrophic organisms (Morgan and Watkinson 1990) or due to ammonia toxicity (Bossert and Bartha 1984) however this is not consistent with the increases in microbial populations found. The decrease in C-min may indicate more efficient oil-C use or bioconversion as was found in other studies (Rimmer and Al-Khafaji 1986, Biederbeck et al. 1993).

6.5 SOIL NUTRIENTS

In this section, the influence of oily waste sludge on nutrient availability, and leaching of oil-related fertilization will be discussed.

6.5.1 Nitrogen

To reduce the amount nutrient leaching, the oil-related fertilizer was applied at three points in time: at oily waste application/incorporation, the following spring prior to soil disturbance and at seeding of the durum and oats. Sludge treated soil, without oil-related fertilization, had consistently lower levels of nitrate-N than the unoiled control soil after one and two years (Table 6.5-1). The amount of total N recovered from the top

Table 6.5-1. Effects of oily waste sludge and oil-related fertilization on nitrate- and ammonium-N in top 10 cm of Hatton loamy sand one and two years after oily waste incorporation.

	Fall 1998		Fall 1999	
Treatment	Nitrate-N	Ammonium-N	Nitrate-N	Ammonium-N
	kg N/ha			
O0U	2.85	1.47	2.62	1.81
O1F	3.50	3.08	4.81	1.89
O1U	2.05	1.81	2.43	1.50
O2F	20.06	14.45	3.28	2.43
O2U	1.40	1.22	1.81	1.63
Analysis of variance†				
Oil	**	**	ns	ns
Oil[Fertilizer]	**	**	*	ns

† ns = not significant; * = significant at p=0.05; ** = significant at p=0.01

10 cm of oiled and fertilized soil after one year is very low considering the large amount applied up to seeding in spring of 1998 (cf. Section 4.3.1). The rapid loss of nitrogen from the treated soil may be related in part to microbial immobilization during decomposition of the oil (McGill et al. 1981) and some losses may have occurred to the atmosphere via the denitrification process but the majority was likely due to leaching of the nitrogen in this loamy sand with poor soil organic matter levels. A single rain event in late June of 1998 (cf. Section 6.1) may have contributed significantly to the leaching during the first field season. Extensive leaching was confirmed by the very high levels of nitrate-N (>165 kg/ha) in soil depths below 30 cm (Figure 6.5-1).

The largest difference between the two years was the dramatic drop in nitrate- and ammonium-N in the O2F treatment (Table 6.5-1). Again reduction in nitrogen levels was attributed to leaching for the most part as levels of nitrate, even at depth, dropped consi-

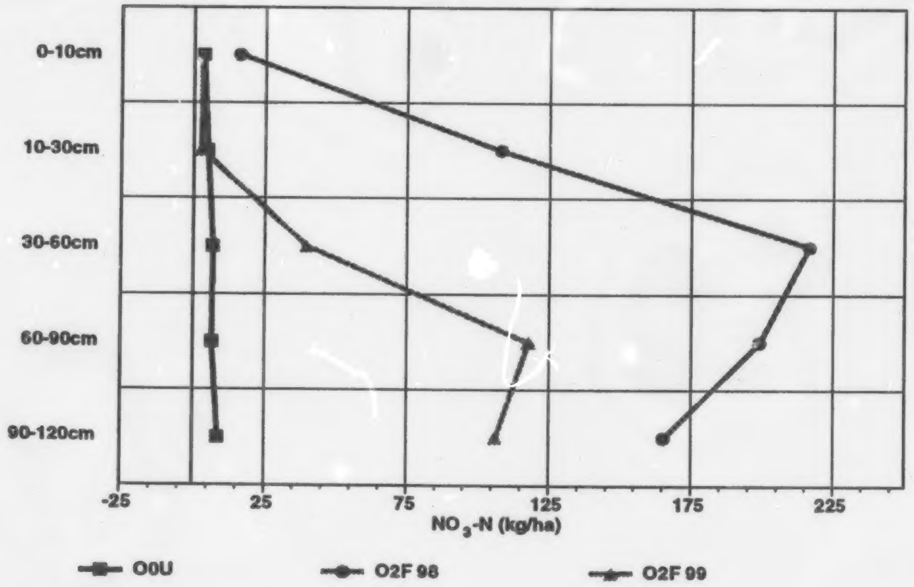


Figure 6.5-1. Nitrate distribution to 1.2 m depth in Hatton loamy sand at one and two years after oily waste application and heavy fertilization.

derably between fall 1998 and fall 1999 (Figure 6.5.1). This loss of nitrogen to below the sampling depth of 120 cm led to the decision to take deep soil cores in the experimental area to determine if the nitrate-N had gone into the groundwater depth. In the spring of 2000, the largest accumulation of soil nitrate-N was at the 1.2 – 1.8 m depth which was still well above the groundwater level, i.e. ~4.0 m depth (Figure 6.5-2).

Ammonium levels in the top 10 cm of soil followed the same pattern as nitrate for the different treatments (Table 6.5-1). Leaching was evident as well for ammonium between fall 1998 and fall 1999 however ammonium-N levels were considerably lower than those of nitrate-N (Figure 6.5-3).

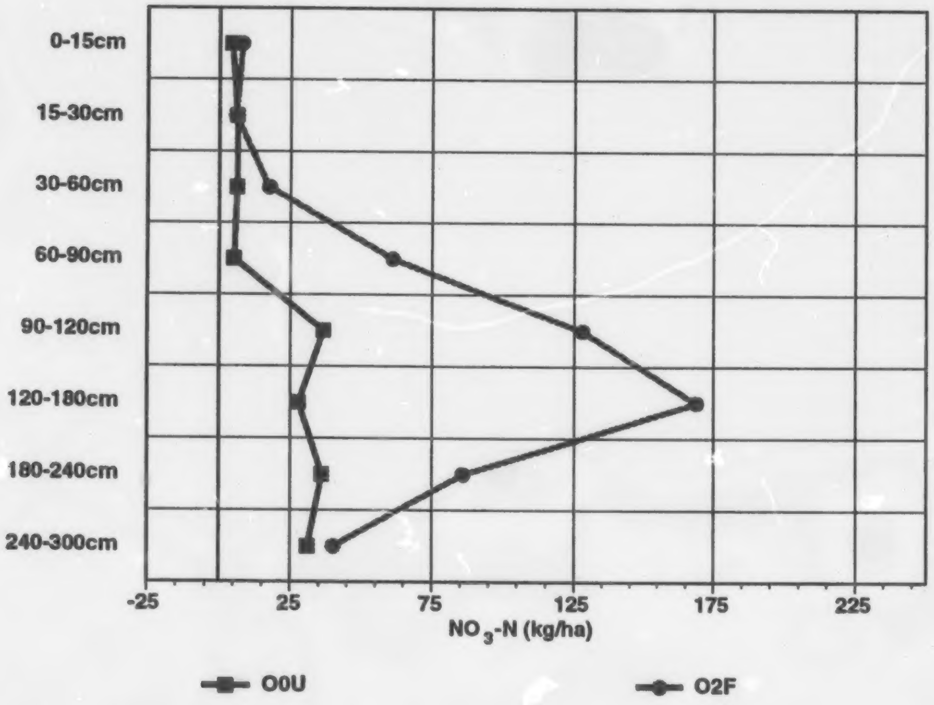


Figure 6.5-2. Nitrate distribution to 3.0 m depth in Hatton loamy sand in spring 2000, i.e. 27 months after oily waste application and heavy fertilization.

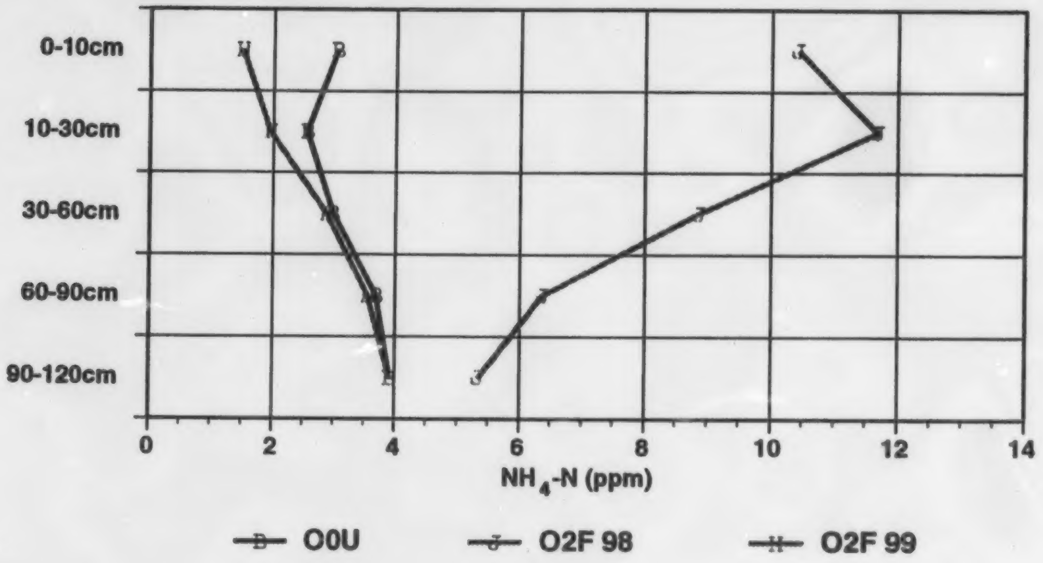


Figure 6.5-3. Ammonium distribution to 1.2 m depth in Hatton loamy sand at one and two years after oily waste application and heavy fertilization.

6.5.2 Phosphate

Plant available (or bicarbonate-extractable) phosphate in the top 10 cm of the Hatton loamy sand varied considerably over the course of the field study (Figures 6.5-4 and 6.5-5). The large reduction in phosphate levels in the top 120 cm from fall 1998 to fall 1999 seem to follow the same trend as the nitrogen (Figure 6.5.4) however in the spring of 2000 the highest level of bicarbonate-extractable phosphate was in the top 10 cm depth. This seems to indicate that oily waste incorporation may have lead to temporary immobilization of phosphate that was later released during microbial biomass turnover.

6.5.3 Sulfur

Sulfate levels in the 90 – 120 cm depth of soil were always higher than that found in the above depths (Figure 6.5-6). Although the 0-10 cm depth sulfate levels were in-

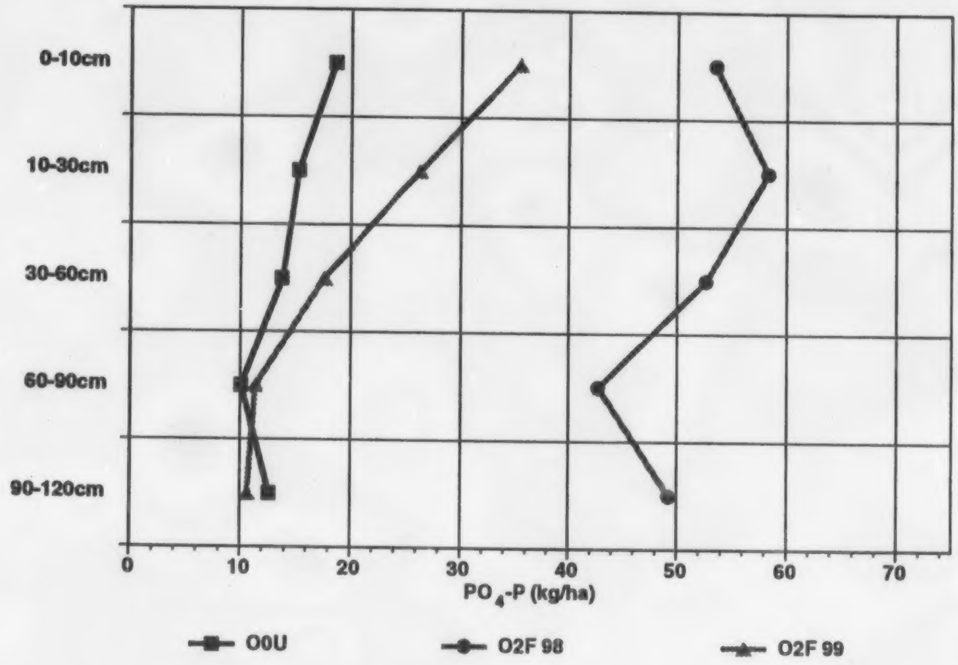


Figure 6.5-4. Phosphate distribution to 1.2 m depth in Hatton loamy sand at one and two years after oily waste application and heavy fertilization.

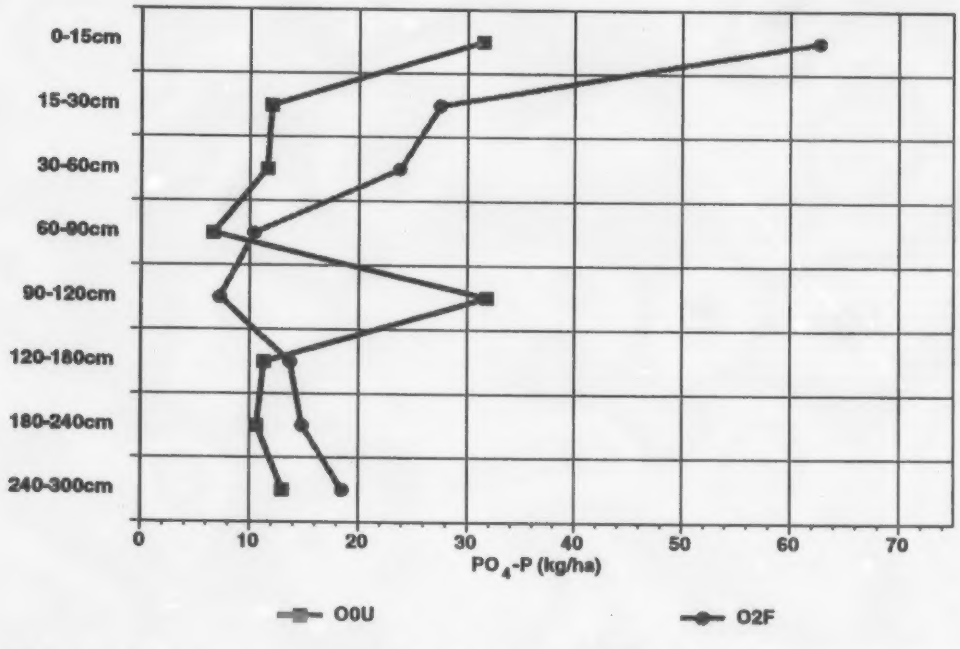


Figure 6.5-5. Phosphate distribution to 3.0 m depth in Hatton loamy sand in spring 2000, i.e. 27 months after oily waste application and heavy fertilization.

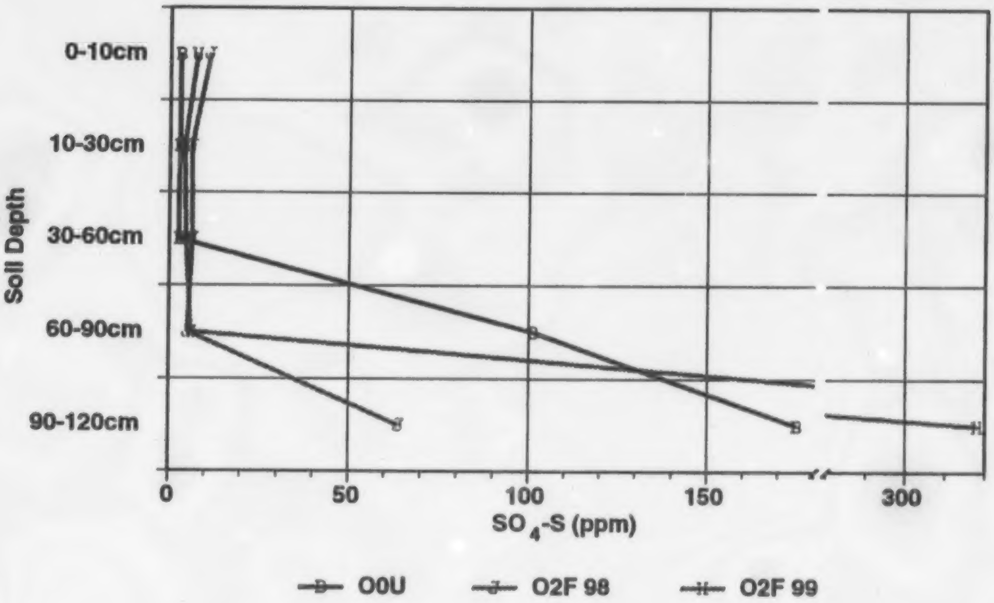


Figure 6.5-6. Sulfate distribution to 1.2 m depth in Hatton loamy sand at one and two years after oily waste application.

creased slightly in 1998 it appears the majority of the sulfate had already leached out of the top 1.2 m. Sandy soils adsorb little sulfate hence why little was retained in the plant rooting depth.

6.5.4 Conclusions

The heavy N & P fertilization of the oiled plots, although split into three separate applications, put much more nutrients than could be used or immobilized by oil-degrading microbes, especially in under the drought stress of spring and summer 1998 or could be physico-chemically absorbed and retained within the sludge-treated layer of Hatton loamy sand. Consequently, one heavy rainfall at the end of June 1998 (cf. Section6.1) contributed greatly to massive leaching of nitrates down to and beyond the

120 cm depth. The nutrient analyses of deep soil cores taken in spring 2000, indicated that nitrate-N levels at depth had not yet reached the level of groundwater at the site.

From the analyses obtained during this study, it is clear that the initial fertilization applications were much too large for the soil to retain at the sludge-treated soil layer. This leads to the conclusion that more frequent applications of oil-related fertilizer are required to ensure adequate nutrient availability for soil microorganisms over the course of the bioconversion process and to limit potential losses to leaching and contamination of groundwater.

6.6 VEGETATIVE PLANT GROWTH

The impact of hydrocarbons in soil on plant growth has been shown to vary considerably (Giddens 1976, Rowell 1977, Mitchell et al. 1988, Danielson et al. 1990, Biederbeck et al. 1993). A great deal of information on phytotoxicity of hydrocarbons has been gained from the use of herbicidal oil (Baker 1970) and also from experience with oil spills (McGill et al. 1981). The effect of hydrocarbons on plants can range from direct effects such as killing the plant on contact and inhibition of seed germination to indirect effects like growth reductions due to changes in soil moisture retention, nutrient availability and development of anaerobic conditions (Rowell et al. 1992), and on to stimulation of growth at low concentration of HC (Baker 1970, Biederbeck and St. Jacques 1988). Phytotoxicity depends not only on the plant type but also on the chemical nature of the applied hydrocarbons. Toxicity generally decreases with an increase in molecular size and viscosity (McGill et al. 1981) therefore fuel oils are more phytotoxic than unrefined oils or heavier sludges.

6.6.1 Cereal crop populations

On April 15, 1998 durum, cv. Kyle, and oats, cv. Calibre, were seeded in to the 48 plots on the west side of the field trial area. The crops were found to be emerging after 16 days and emergence counts were taken periodically thereafter. The seeding rate used for both crops should have resulted in emergence of approximately 200 plants within one square metre. Oil application/incorporation and oil-related fertilization were shown to be detrimental to crop emergence from the first emergence count date (Figure 6.6-1). With

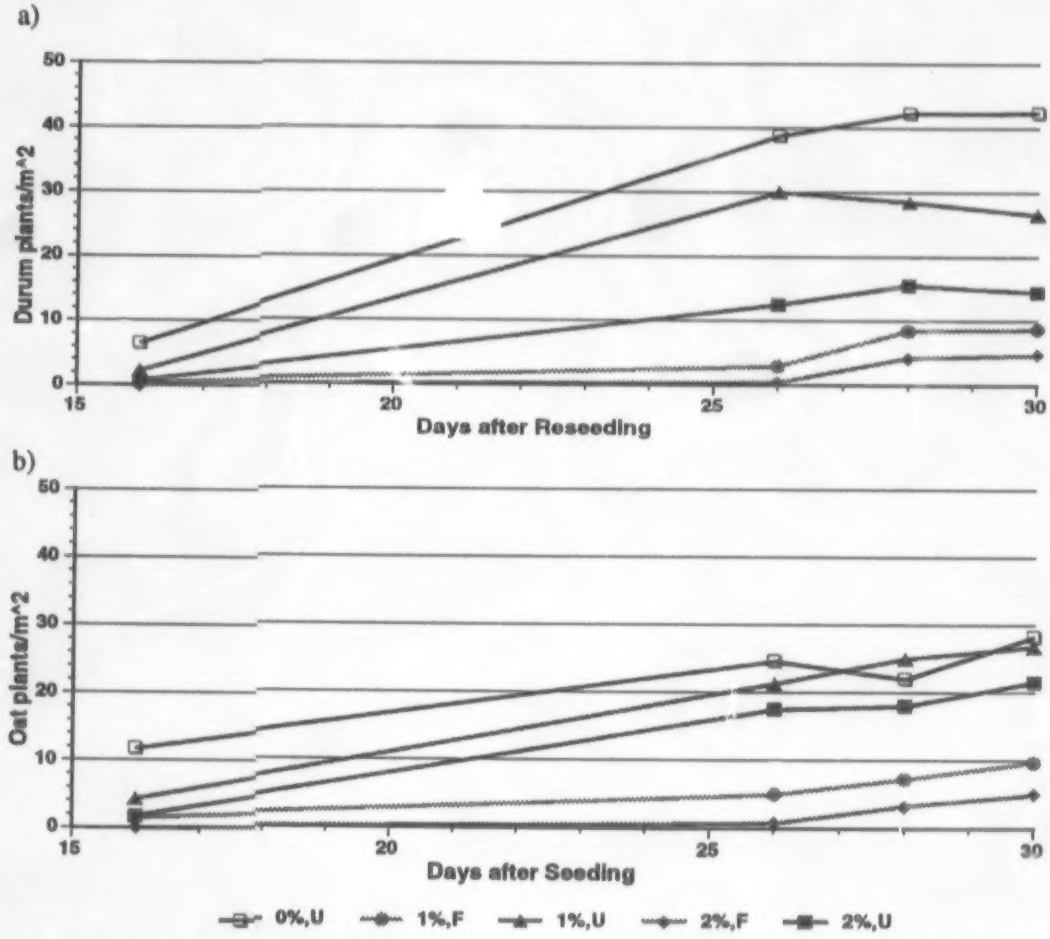


Figure 6.6-1. Emergence counts of a) Kyle durum and b) Calibre oats at the field site near Richmound, SK after seeding on April 15, 1998.

less than one quarter of the seeded crop emerged after 30 days, even in the control plots (Figure 6.6-1), it was decided that the site should be reseeded. On May 27, 1998 the previously cropped area was sprayed with Round-Up and then later that day reseeded to the same crops at the same rates. The crops were found to have emerged after 20 days and emergence counts were taken periodically thereafter. Oily waste application had less effect on the emergence of the crops at this time than did the oil-related fertilization for both crops especially the oats (Figure 6.6-2). By 40 days after reseeding both crops had reached at least 70% of the maximum emergence possible at the seeding rates used. This was a large improvement with all treatments having at least a 3-fold increase in plant numbers compared to the earlier seeding.

On May 4, 1999 the east side of the field site (fallow in 1998) was seeded to Kyle durum and Calibre oats at the same rates as the previous year. The crops were found to be emerging after 16 days and emergence counts were taken periodically thereafter. At 27 days the emergence counts were approaching 50% of the possible maximum emergence, however after this date the crop stand went down (Figure 6.6-3). The reason for the reduction in plant numbers is uncertain however similar observations were found by Biederbeck et al. (1993). Kyle durum was found to be less affected by the oily waste sludge from 27 days after seeding on compared to the Calibre oats (Figure 6.6-3). The reason for the difference observations between 1998 and 1999 emergence counts is uncertain.

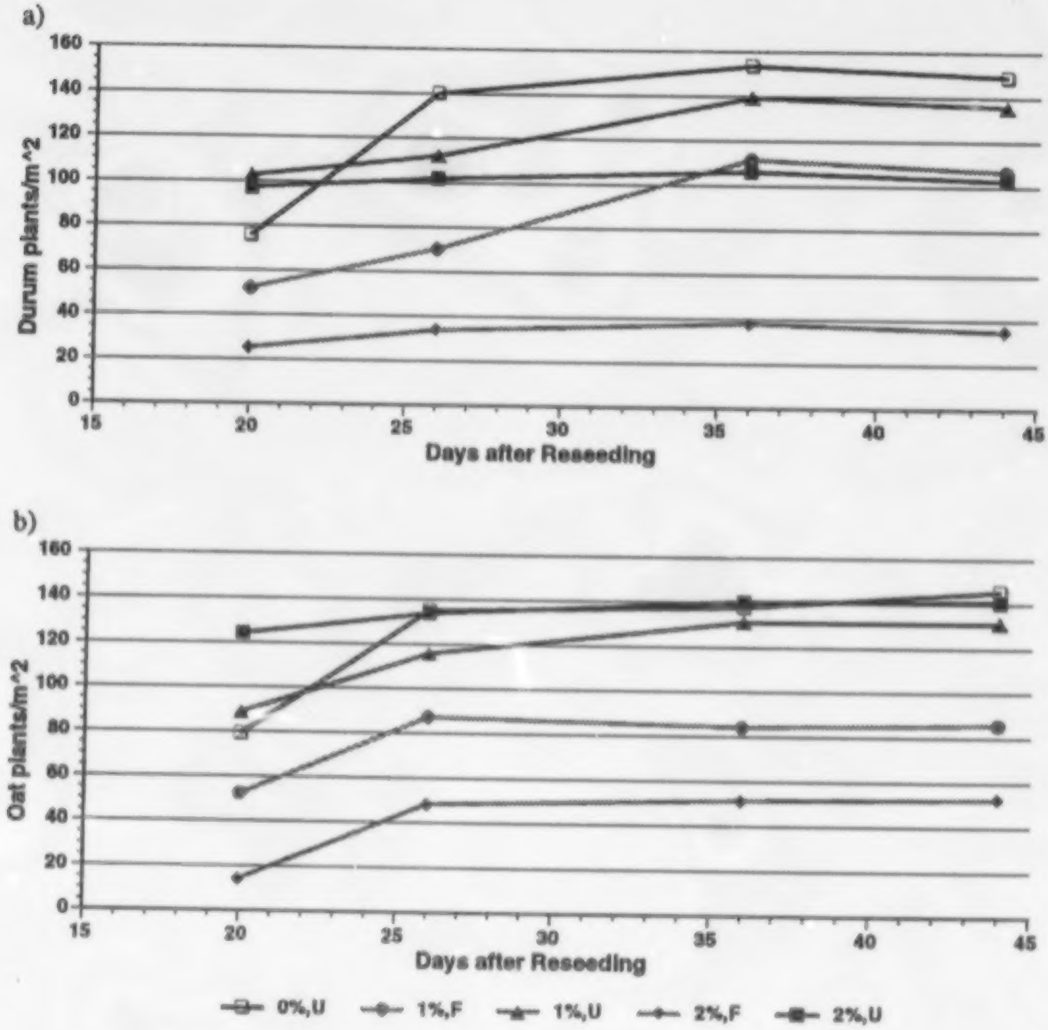


Figure 6.6-2. Emergence counts of a) Kyle durum and b) Calibre oats at the field site near Richmound, SK after reseeding on May 27, 1998.

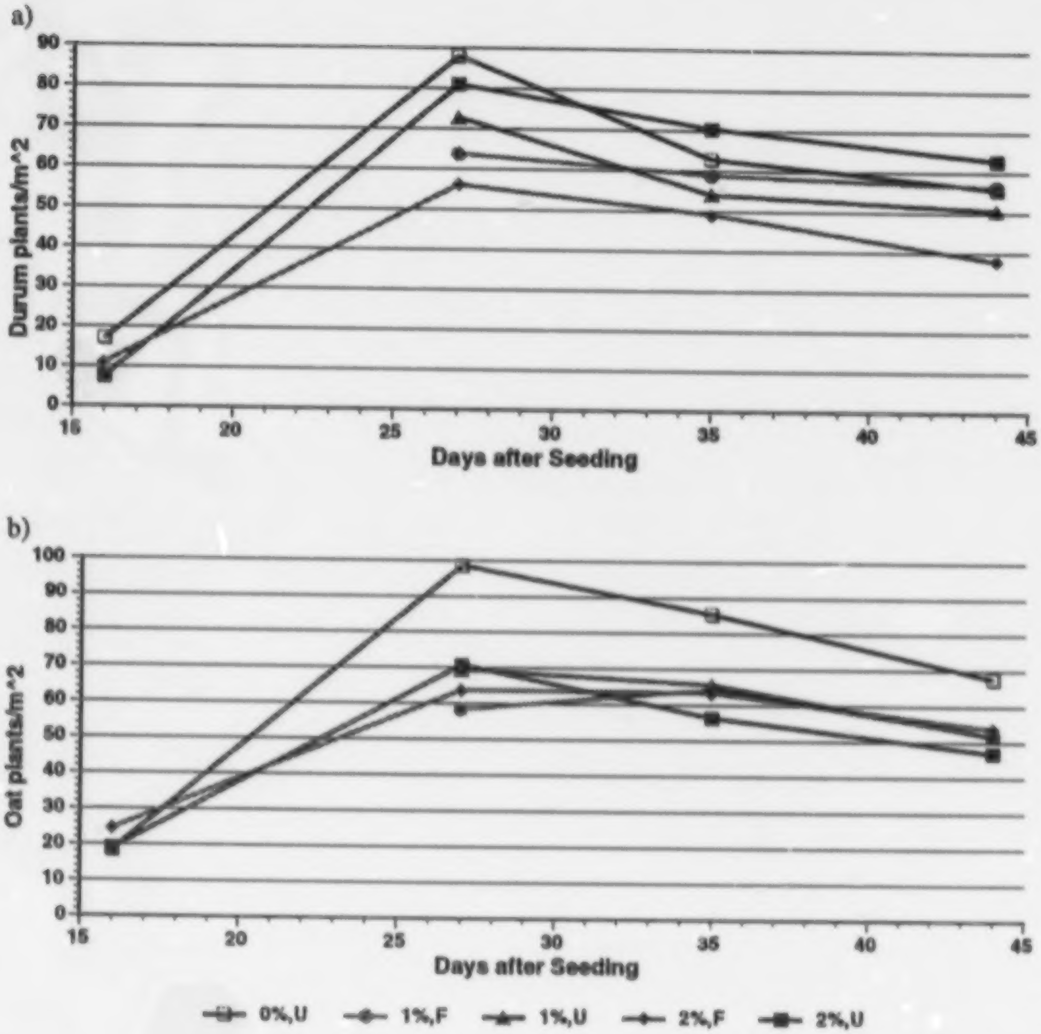


Figure 6.6-3. Emergence counts of a) Kyle durum and b) Calibre oats at the field site near Richmound, SK after seeding on May 5, 1999.

6.6.2 Cereal crop population and biomass at anthesis

At the end of July in 1998 and 1999, when the crop was in anthesis (i.e., flowering), square metre samples were cut in all plots to determine stand density and above-ground dry matter biomass. Crop populations were two- to three-fold greater in

1998 than in 1999 (Figures 6.6-4 and 6.6-5). The reason for the reduction in plant populations are uncertain especially due to the more favourable moisture conditions in the early portion of the growing season in 1999 compared to the limited precipitation in May of 1998. Any oily waste phytotoxicity should have been reduced over the previous 18 months after sludge incorporation prior to seeding in May of 1999 as the oily materials were modified by biodegradation (Biederbeck et al. 1993) however it may be possible that biodegradation products that inhibit seed germination were present during this portion of the field season.

The population decrease in 1999, compared to 1998, was not reflected in the biomass of the crops at anthesis. Generally, large increases in biomass were found in the oiled treatments especially those with oil-related fertilization (Figures 6.6-6 and 6.6-7). Oil-related fertilization seemed to further reduce crop dry matter production in 1998 at anthesis. A notable exception to this is durum with treatment O1U, which was greatly impacted by oil application and yet fertilization with 1% oil (O1F) returned the biomass to equal that of the control.

From the large increases in dry matter produced by anthesis in 1999, compared to the 1998 field season, it seems apparent that any phytotoxicity present which effected plant growth once the plant was established during the first field season must have been greatly reduced. Kyle durum biomass was clearly increased by the addition of oily waste and further boosted by fertilization (Figure 6.6-7). Calibre oats showed slightly reduced dry matter production in oiled soil and greater reduction with oil-related fertilization. Crop response differences to oil and fertilizer are evident for both years at anthesis.

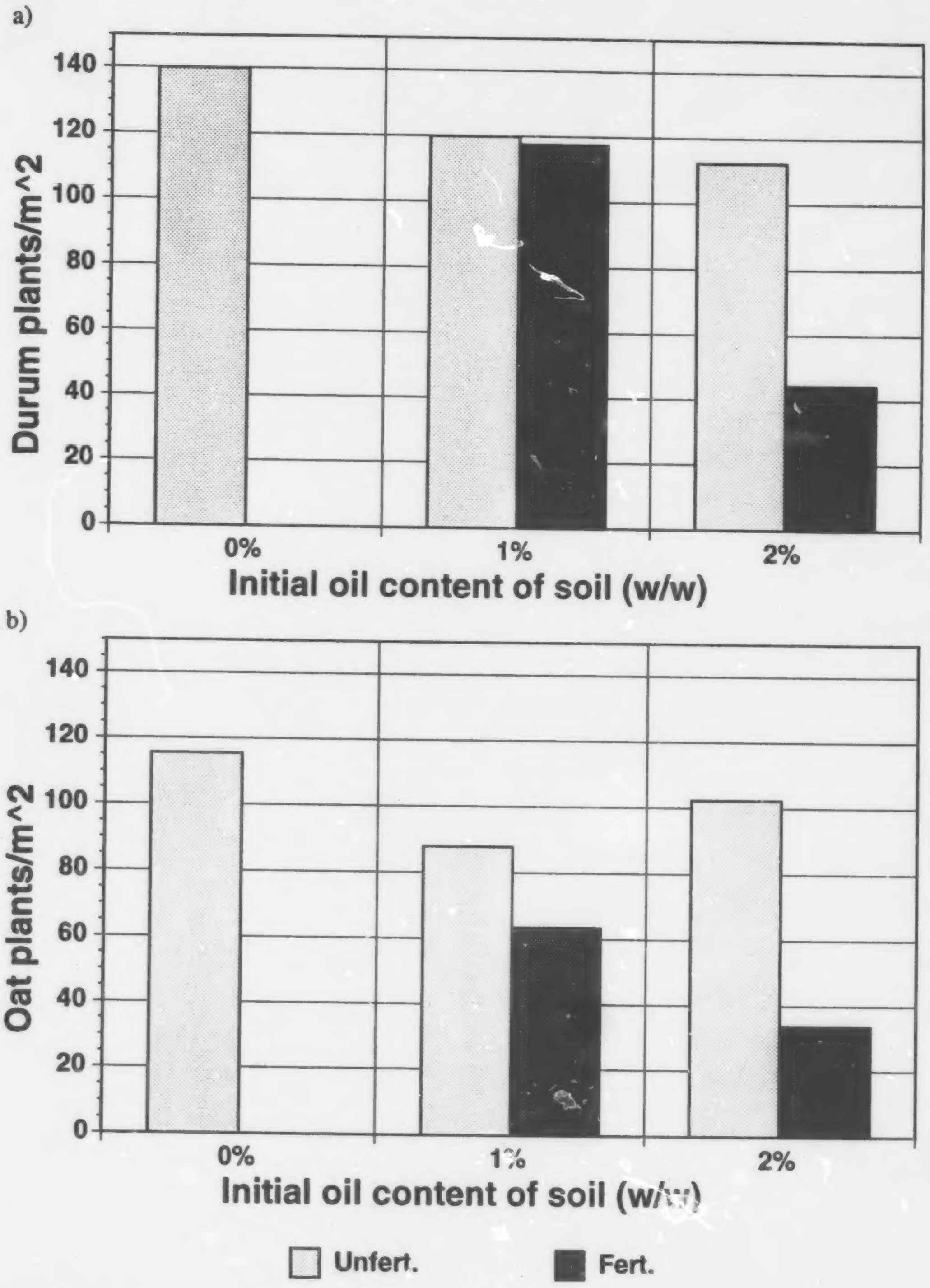


Figure 6.6-4. The effect of incorporation of oily waste sludge and fertilization on populations of a) Kyle durum and b) Calibre oats at anthesis in 1998.

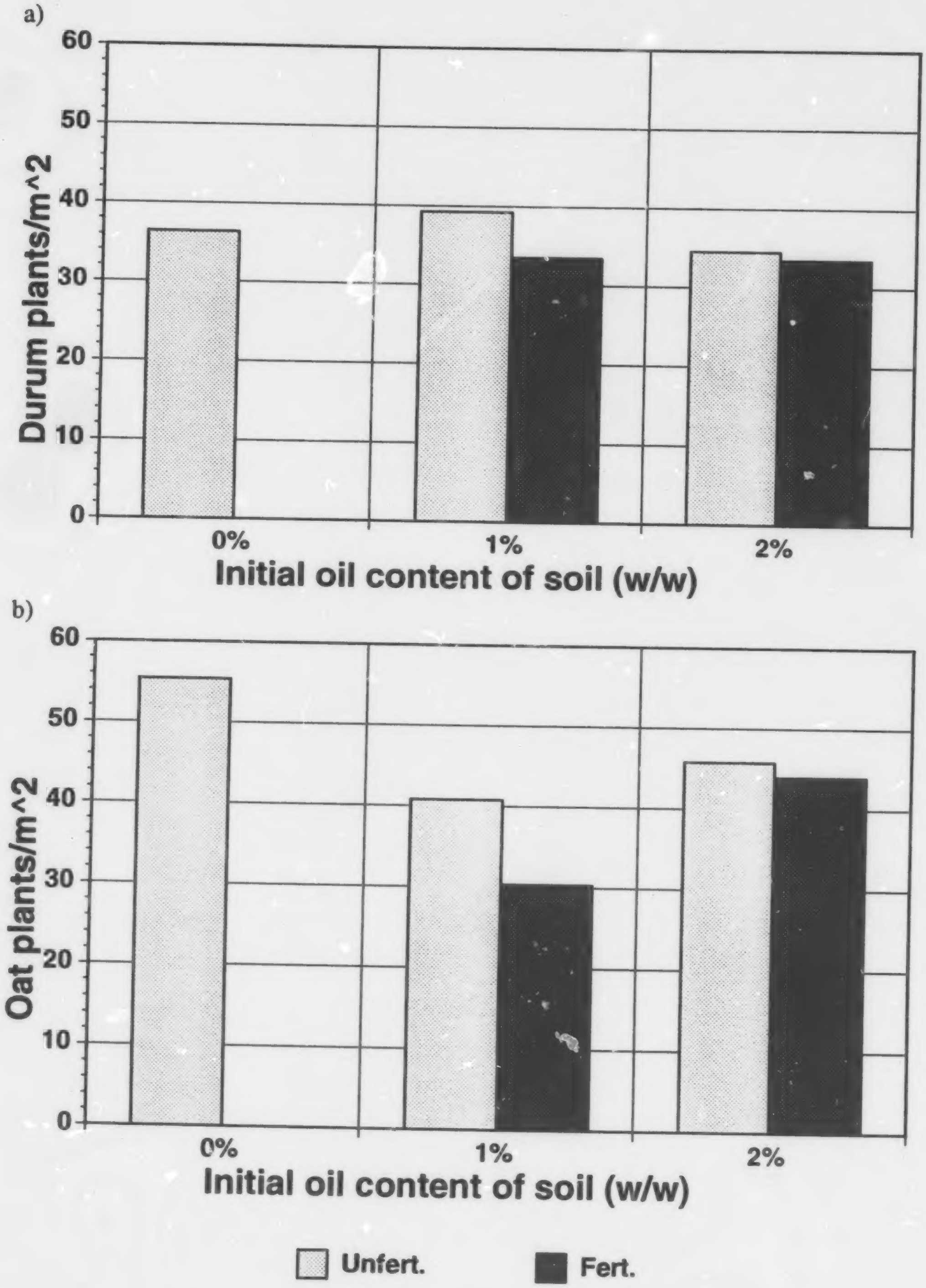


Figure 6.6-5. The effect of incorporation of oily waste sludge and fertilization on populations of a) Kyle durum and b) Calibre oats at anthesis in 1999.

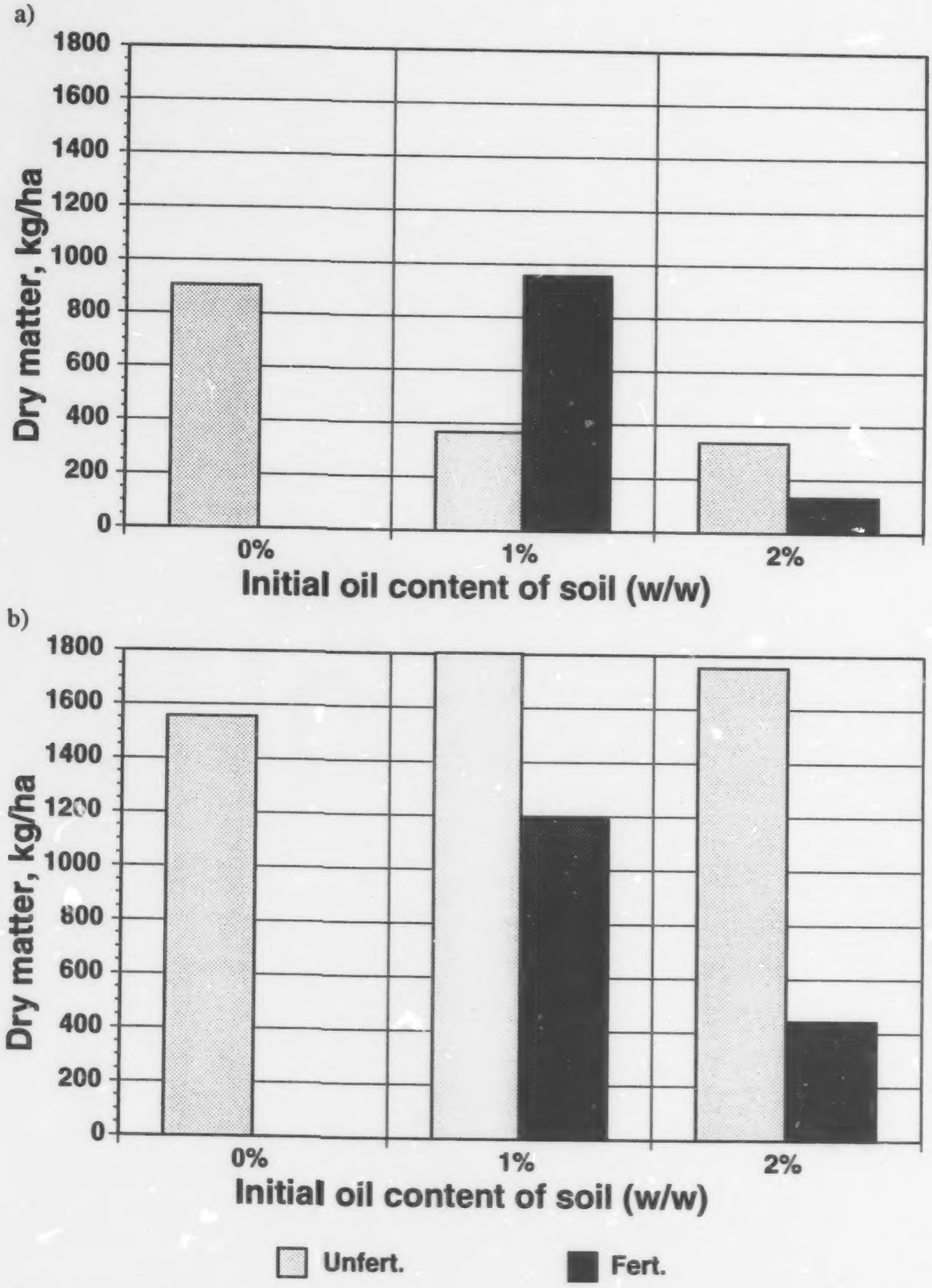


Figure 6.6-6. The effect of incorporation of oily waste sludge and fertilization on dry matter of a) Kyle durum and b) Calibre oats produced up to anthesis in 1998.

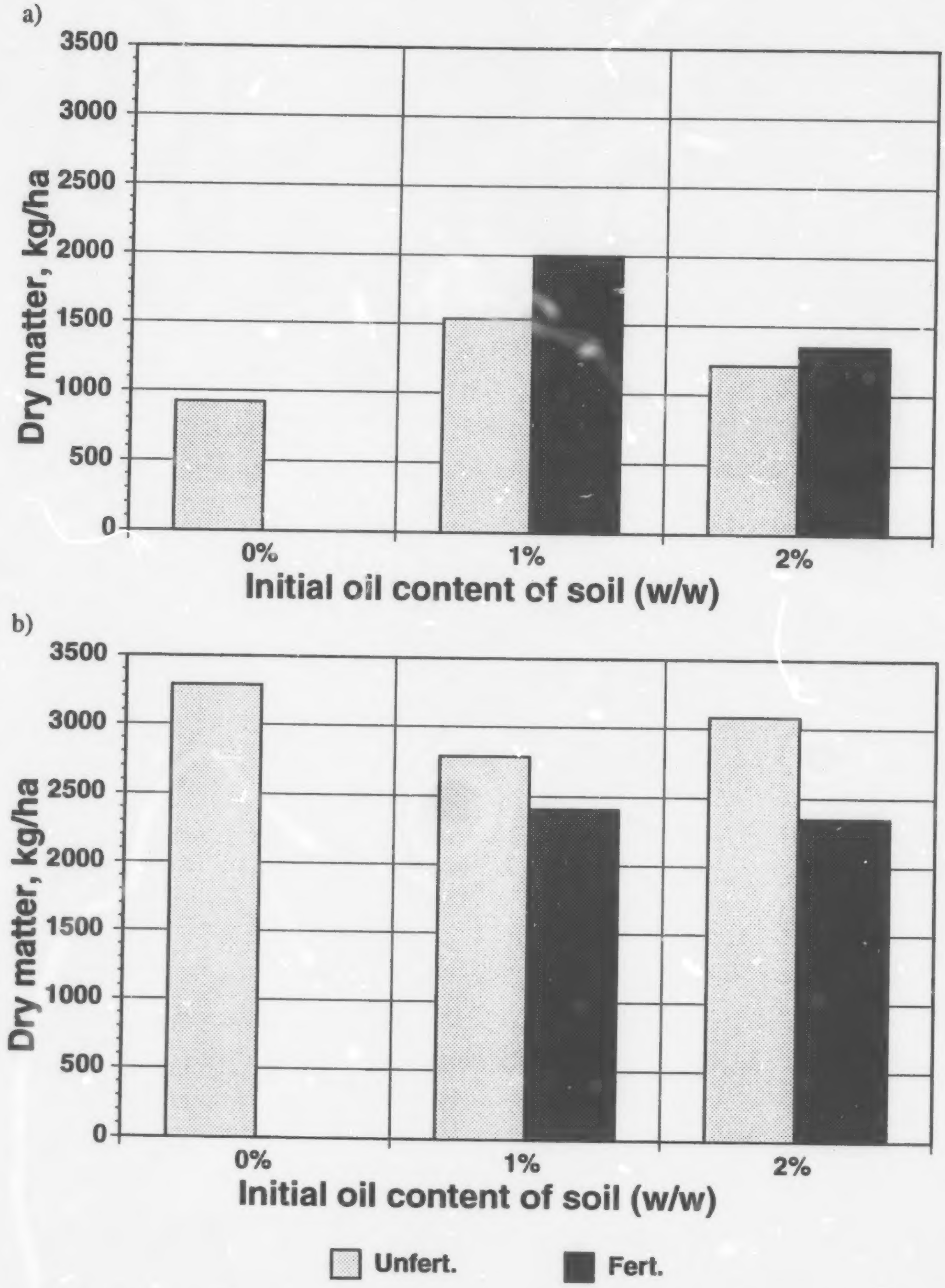


Figure 6.6-7. The effect of incorporation of oily waste sludge and fertilization on dry matter of a) Kyle durum and b) Calibre oats produced up to anthesis in 1999.

6.6.3 Weed populations

Weed counts were initiated at the same time as cereal crop emergence counts in the spring of 1998. The major weed at this site was identified as russian thistle (*Salsola pestifer* A. Nels.). Weed counts were conducted by counting the russian thistle, other broadleaf weeds and grassy weeds. The herbicidal effect of oily waste sludge incorporated to the soil was evident immediately with some 2% oiled plots having over a 25-fold decrease in russian thistle populations compared to some of the unoiled plots (data not shown). The huge numbers of russian thistle that were emerging in the unoiled plots and in the areas between experimental plots however soon became a cause for concern relating to future work at the site. It was then decided that this portion of the study should be terminated and herbicide had to be applied to the site to control the problem weeds.

6.6.4 Elemental composition of cereal crops

Oily waste sludge alone had little or no effect on the nitrogen content of durum and oats at anthesis in 1998 (Figure 6.6-8). This suggests that extensive microbial immobilization of nitrogen, as suggested by McGill et al. (1977), did not occur thus allowing nitrogen to be available to the crop. Oil-related fertilization greatly increased nitrogen content in both crops, especially Calibre oats (Figure 6.6-8). There were significantly greater amounts of soil nitrate- and ammonium-N in the oiled and fertilized plots than the plots that only received oil after harvest in 1998 (cf. Section 6.5.1). This correlates well with the higher levels of N in the plant material.

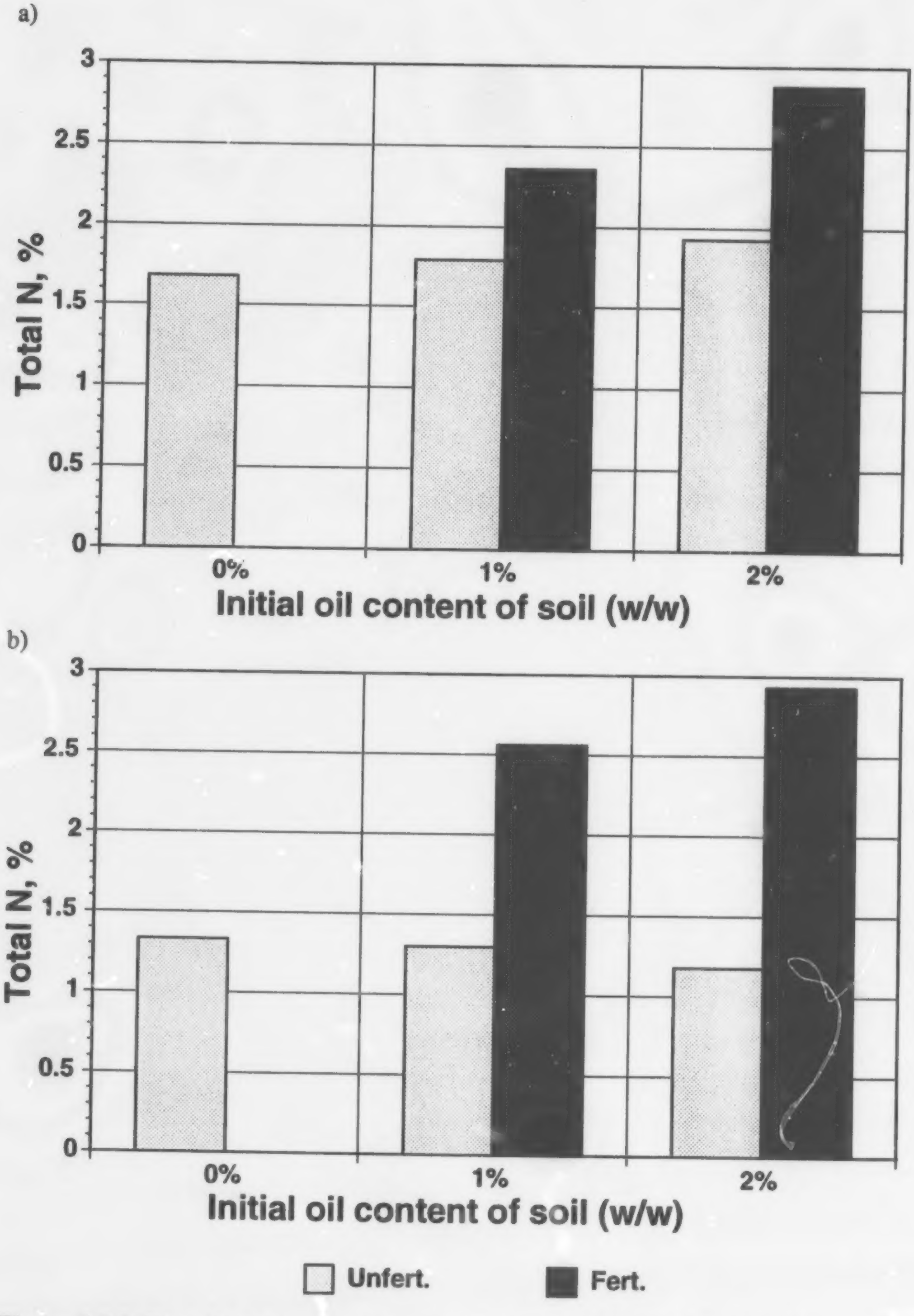


Figure 6.6-8. The effect of incorporation of oily waste sludge and fertilization on nitrogen content of a) Kyle durum and b) Calibre oats at anthesis in 1998.

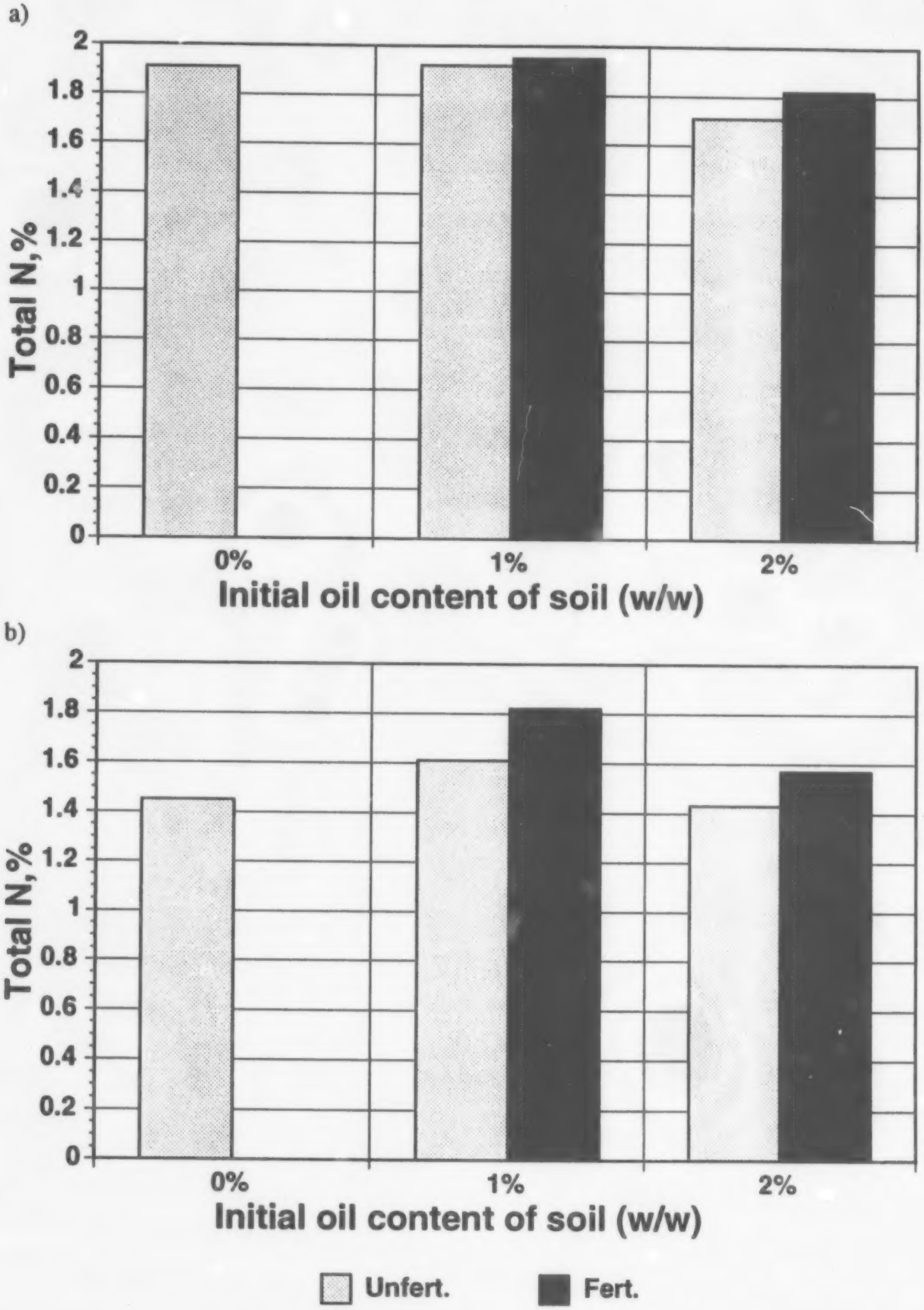


Figure 6.6-9. The effect of incorporation of oily waste sludge and fertilization on nitrogen content of a) Kyle durum and b) Calibre oats at anthesis in 1999.

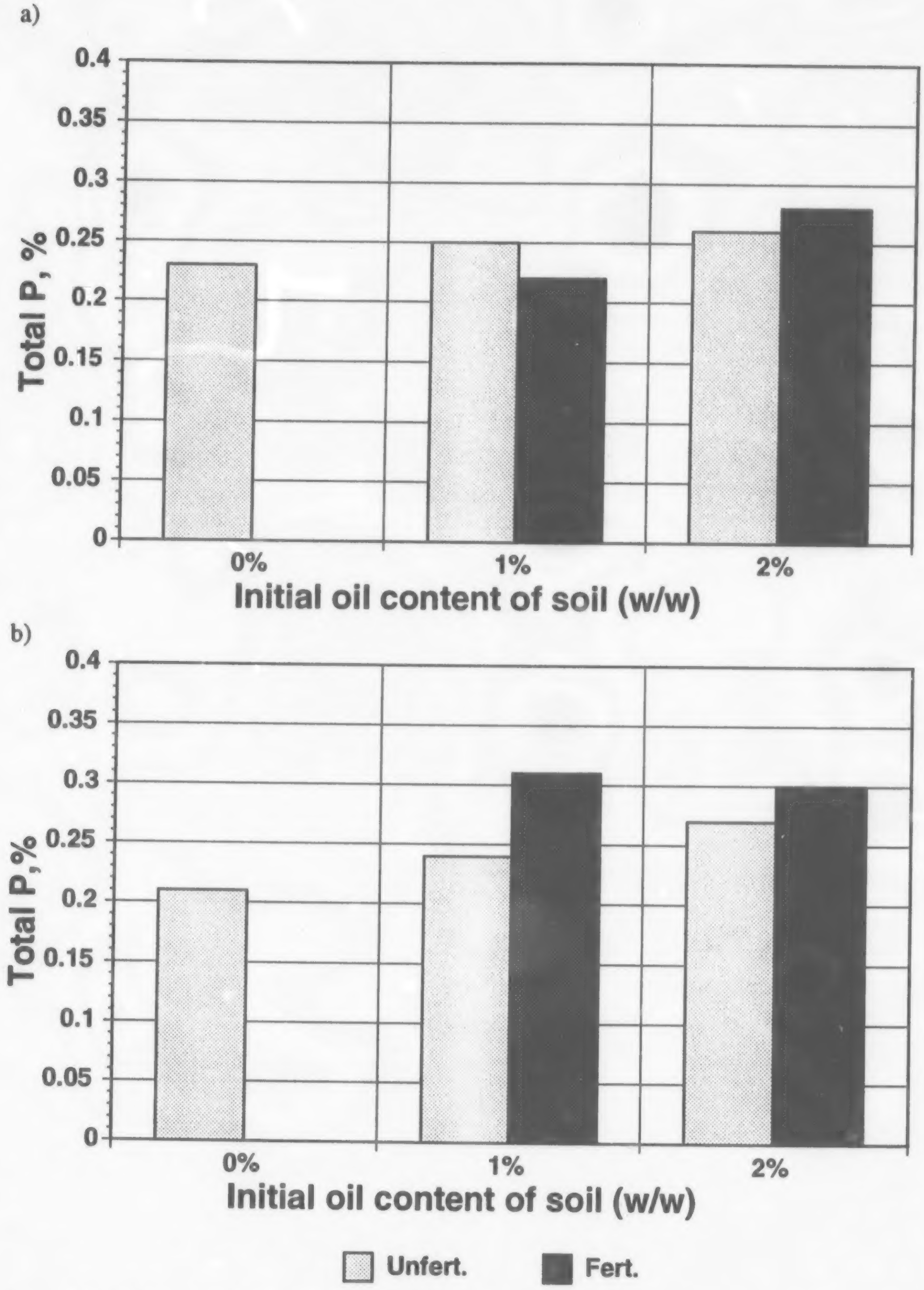


Figure 6.6-10. The effect of incorporation of oily waste sludge and fertilization on phosphorus content of a) Kyle durum and b) Calibre oats at anthesis in 1998.

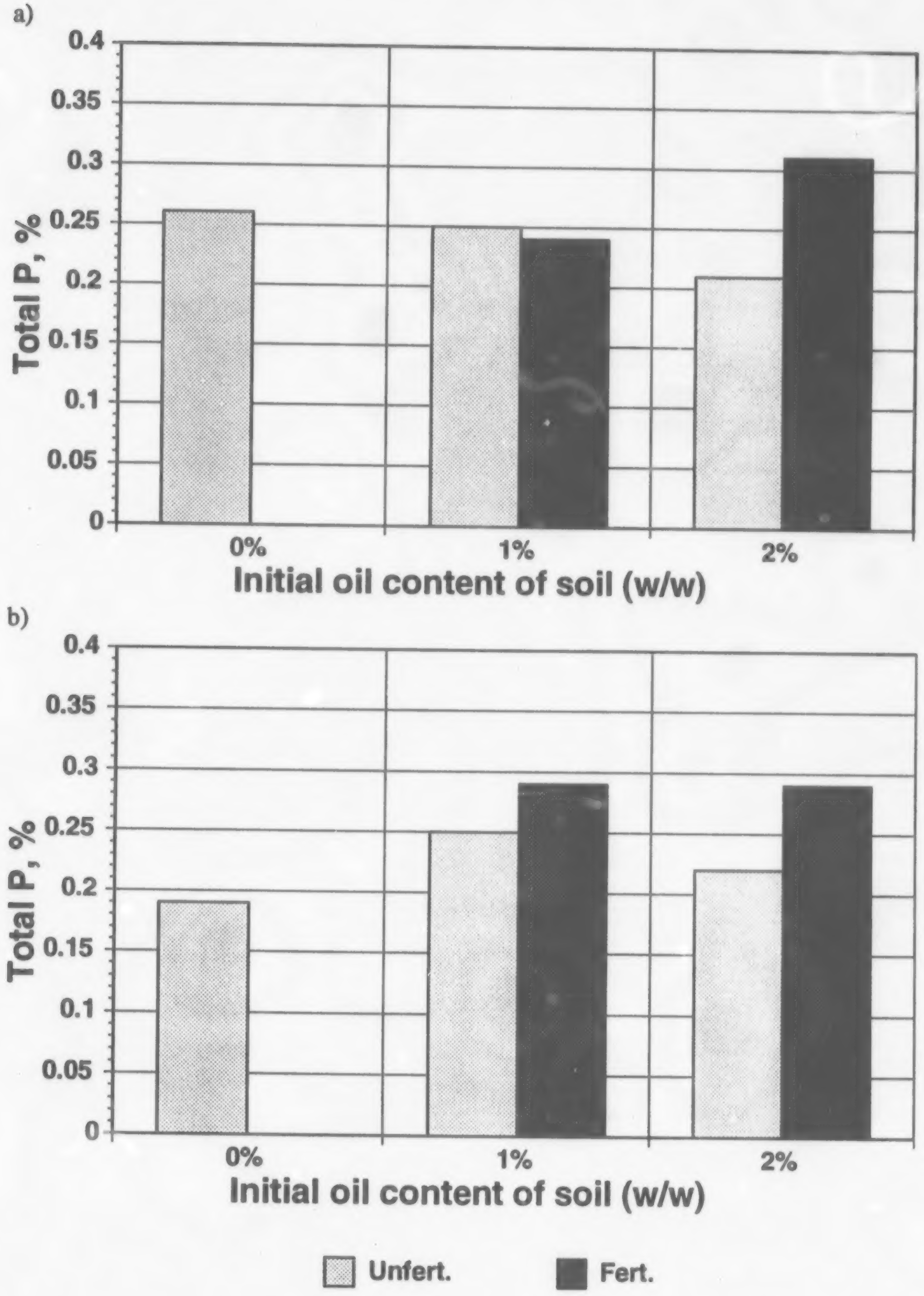


Figure 6.6-11. The effect of incorporation of oily waste sludge and fertilization on phosphorus content of a) Kyle durum and b) Calibre oats at anthesis in 1999.

6.7 YIELD AND QUALITY OF GRAIN

Crops are not produced in industrial 'landfarming' systems because the land area is subjected to frequent tillage and other bioremediation techniques (Rowell et al. 1992). The main objective of this study was to improve marginal cropland thus the effect of oily waste sludge incorporation on grain yield and quality must be negligible or even an improvement to aid adoption of this methodology.

6.7.1 Yield of grain

Due to the drought conditions in 1998, crop production in unoiled plots was severely reduced (Figure 6.7-1). Many of the 2% oiled plots, especially those that received oil-related fertilization, had no growth and if there was growth the crop was so short that harvesting mechanically was difficult (Figure 6.7-2).

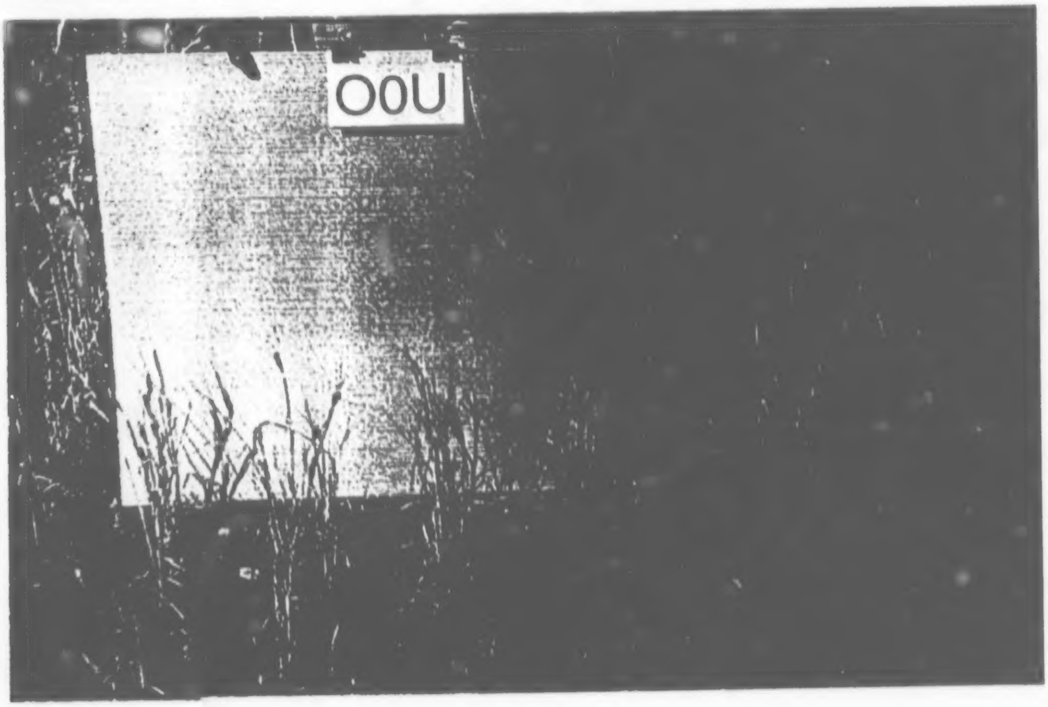


Figure 6.6-1. Stand of Kyle durum at harvest in an unoiled control plot in August 1998.

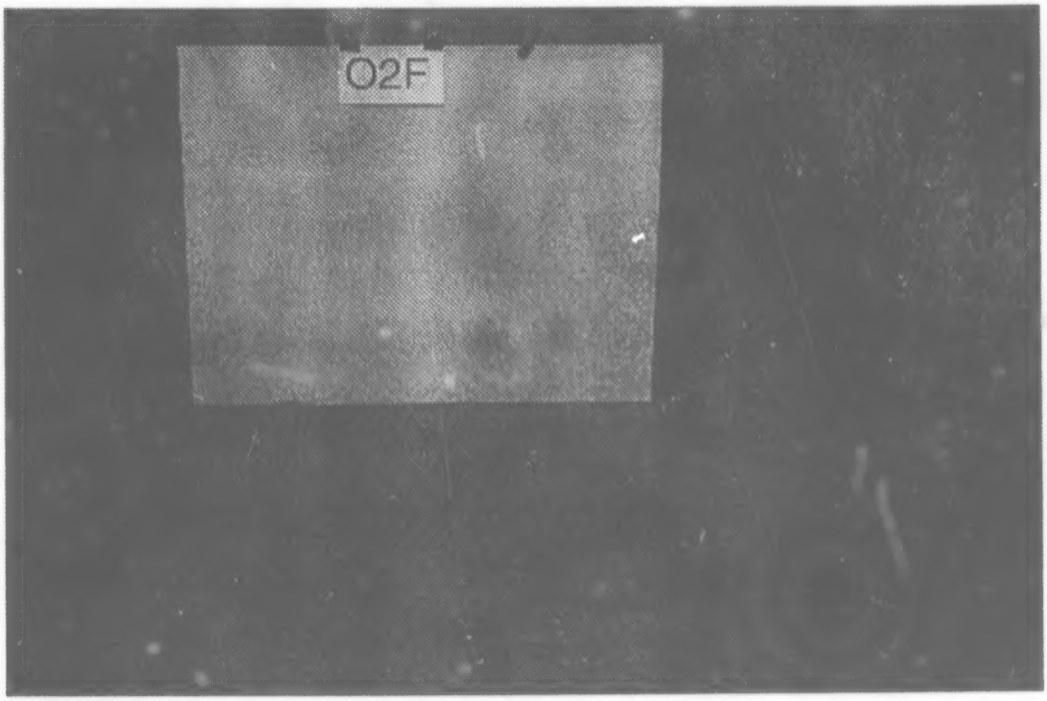


Figure 6.7-2. Stand of Calibre oats in the 2% oiled treatments unfertilized (above) and fertilized (below) at maturity just prior to harvest in August 1998.

The heavy oil-related fertilization, in combination with the sludge application, appeared to have a severely detrimental effect on the crops' ability to establish and hence reach maturity. Due to the drought compounding any crop responses to the oil and fertilizer applications the harvest results for 1998 were deemed too variable and thus unreliable to draw any conclusions. Field studies with barley have shown that oil-induced inhibitions can be severely aggravated by drought and Mitchell et al. (1979) have suggested that the hydrophobicity and phytotoxicity of soil-incorporated oils may be particularly detrimental in dry years.

The much more favourable growing conditions during the 1999 field season allowed the collection of harvest data from all plots. The growth was visibly improved for both durum and oats (Figure 6.7-3).The 'herbicidal' effect of the oily waste was also still evident with very limited weed growth in the oiled plots with pressure from a heavy infestation of wildoats in the north end of the cropped area (Figure 6.7-3). This lack of weed pressure, combined with the soil improvements due to oil incorporation, led to large increases in yield for durum and oats (Figure 6.7-4). Even without the earlier oily waste-related heavy fertilization durum yields were significantly increased by waste sludge application, exceeding check yields by 40% at the 1% initial soil oil content and by 80% at the 2% soil oil content (Figure 6.7-4a). The residual benefit of the oil-related fertilization was reflected in further increases to durum yield especially the O1F treatment that resulted in a 160% higher yield than the check.

Oat yields were also significantly raised by oily waste application/incorporation alone with increases of about 30% and 50% for the 1 and 2% soil oil treatments, respectively (Figure 6.7-4b). Again the oil-related fertilization further enhanced the amount of grain harvested by adding approximately 20% more yield for both soil oil levels compared to the control. The very large increases in crop yields suggests that the

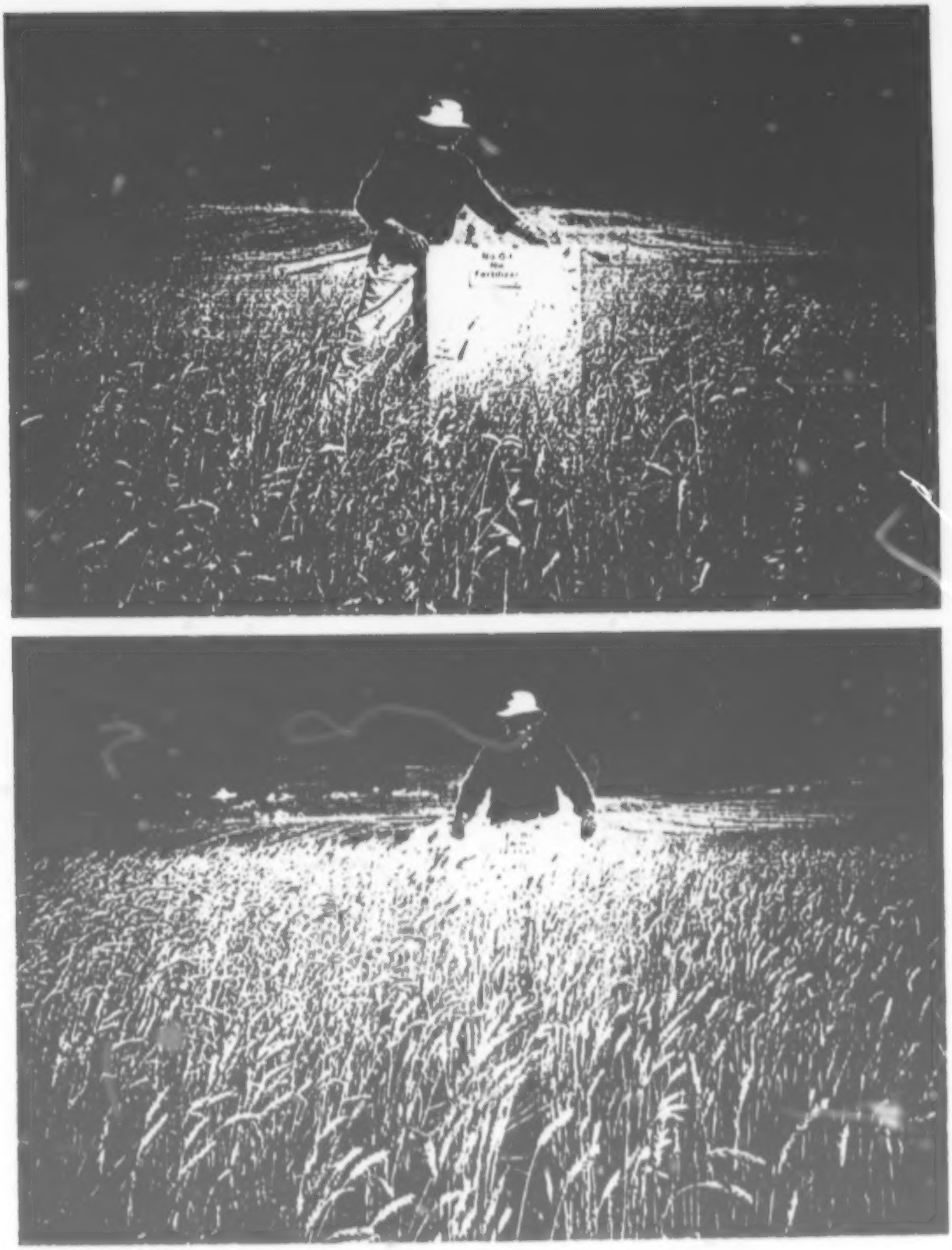


Figure 6.7-3. Stand of Kyle durum in the check plot, O0U (above) and 2% oiled fertilized, O2F (below) at maturity just prior to harvest in early September 1999. Note heavy wildoat infestation in the unoiled check.

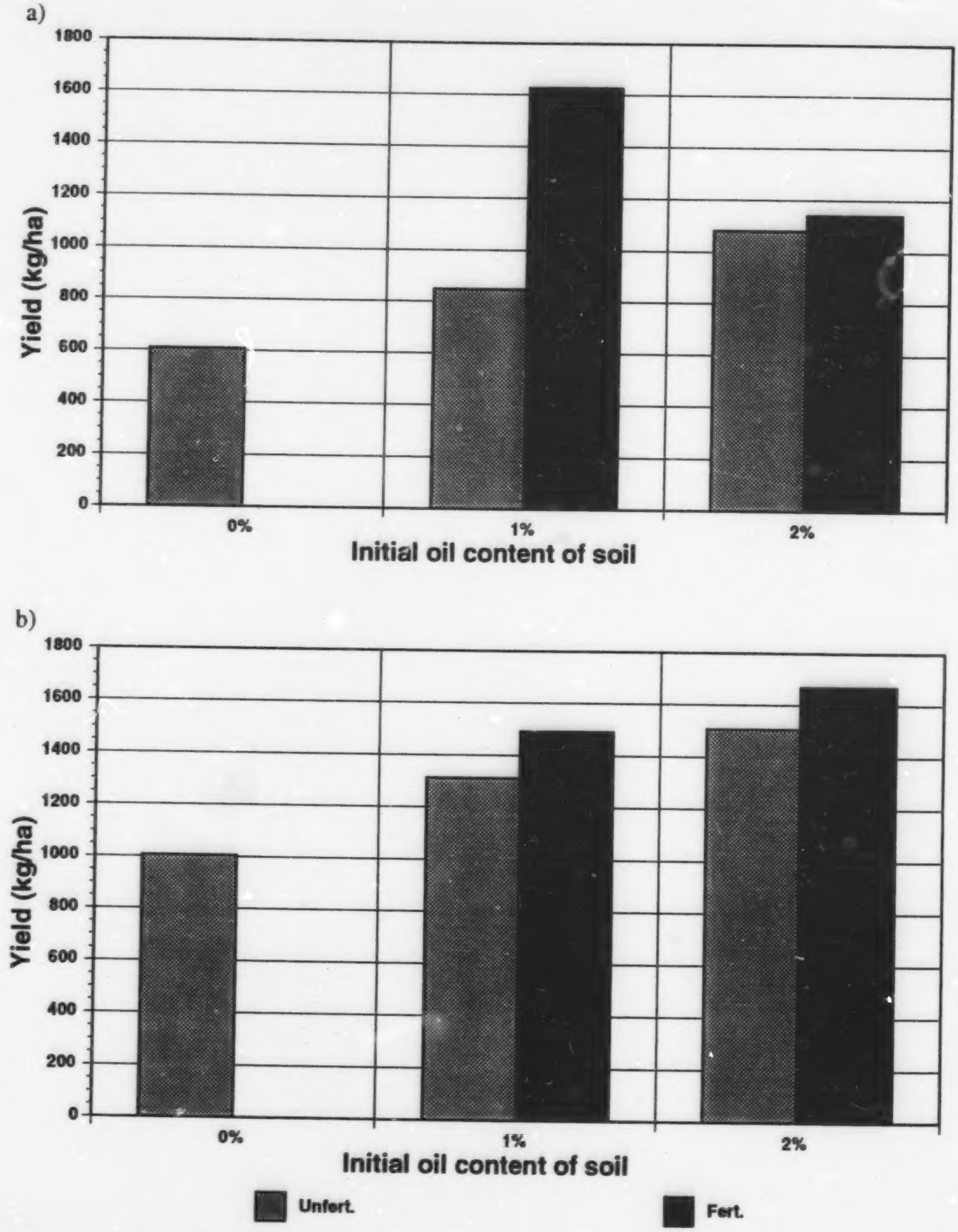


Figure 6.7-4. Effect of oily waste sludge incorporation and oil-related fertilization on grain yield of a) durum, cv. Kyle, and b) oats, cv. Calibre, in 1999.

CCRL oily waste sludge either had low phytotoxicity for the crops chosen or that the detrimental components in the sludge were biodegraded/converted during the previous year thus resulting in only beneficial effects on crop growth for the 1999 field season.

Another interesting effect that may be related to benefits of oil incorporation and oil-related fertilization in agricultural soils is the increase in the number of heads per plant (Figure 6.7-5). Both crops showed modest (7%) to very substantial (75%) increases in the number of heads per plant compared to the controls. The effect was not consistent for oil application alone thus it appears that fertilization may be the most important factor contributing to the observed increases.

6.7.2 Grain quality

The indices used to evaluate the effects of sludge on grain quality included: test weight (hectolitre weight), kernel weight (weight per 1000 kernels), and protein and phosphorus content. In addition, selected grain samples were analyzed for heavy metals at the Dept. of Soil Science, University of Saskatchewan.

Oil and fertilizer treatments had little effect on the hectolitre weight of durum (Figure 6.7-6a). However, oat test weights were depressed at 1% oil with or without fertilization while the results at 2% oil were variable (Figure 6.7-6b). Crop responses to the treatments were similar for 1000 kernel weight with little effect on durum and results for 1% oil in oats slightly depressed (Figure 6.7-7).

Considering the very large increases in crop yield (cf. Section 6.7.1) the amount of protein in the grain remained very high with some treatments even showing slight increases compared to the control (Figure 6.7-8). Fertilization generally increased protein levels more than oil alone, presumably due to the additional supply of nitrogen available

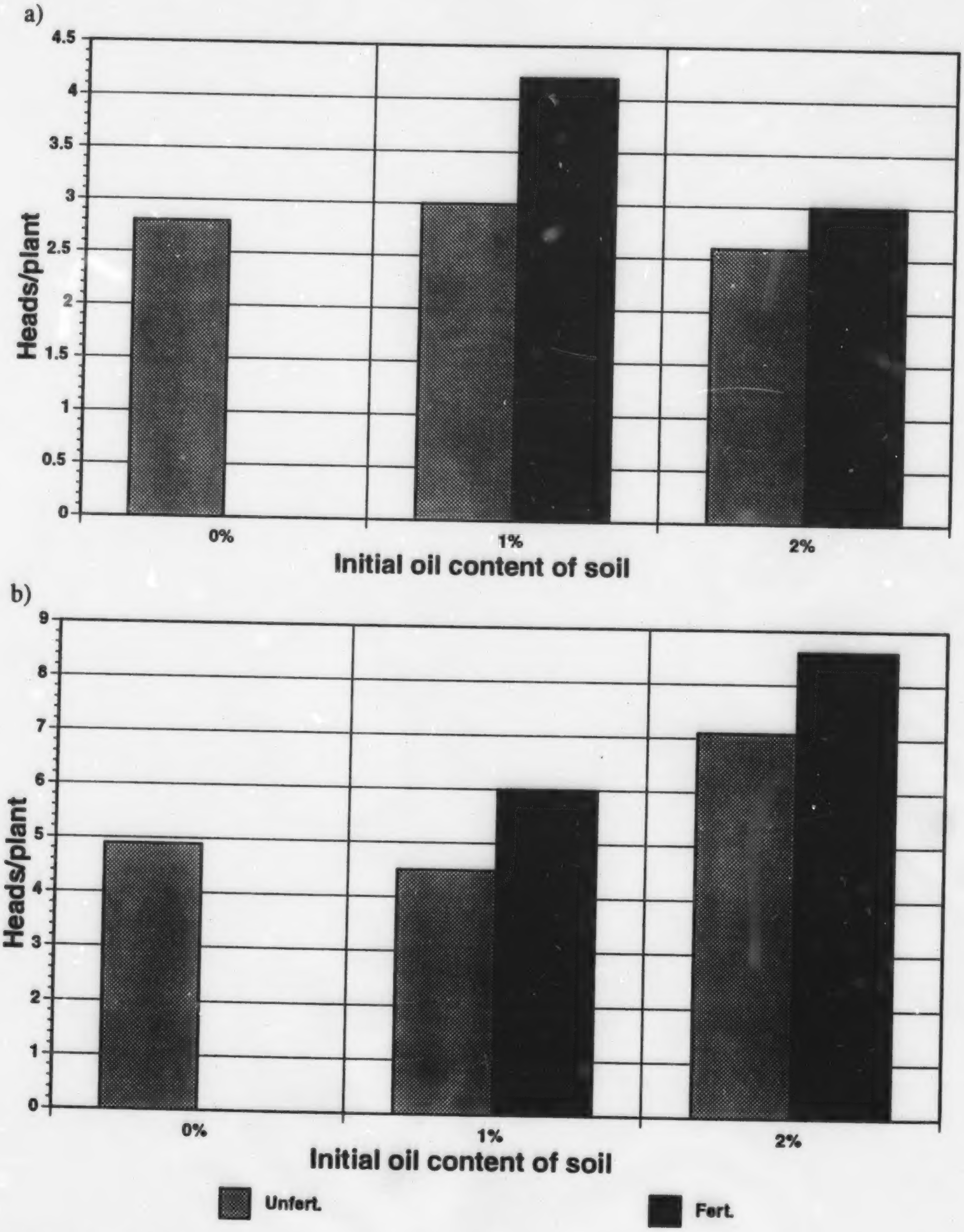


Figure 6.7-5. Effect of oily waste sludge incorporation and oil-related fertilization on the number of heads per plant of a) durum, cv. Kyle, and b) oats, cv. Calibre, in 1999.

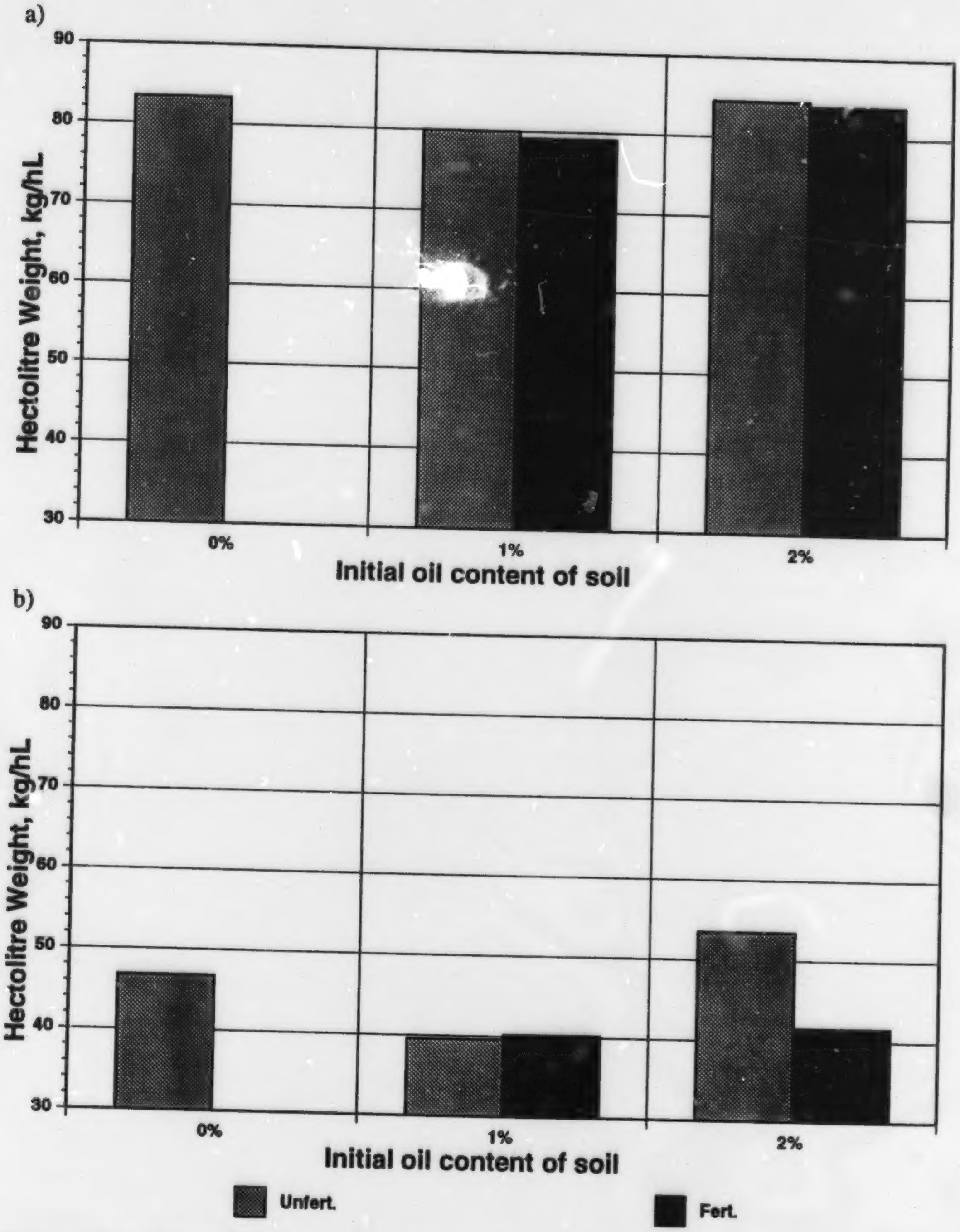


Figure 6.7-6. Effect of oily waste sludge incorporation and oil-related fertilization on hectolitre weight of a) durum, cv. Kyle, and b) oats, cv. Calibre, in 1999.

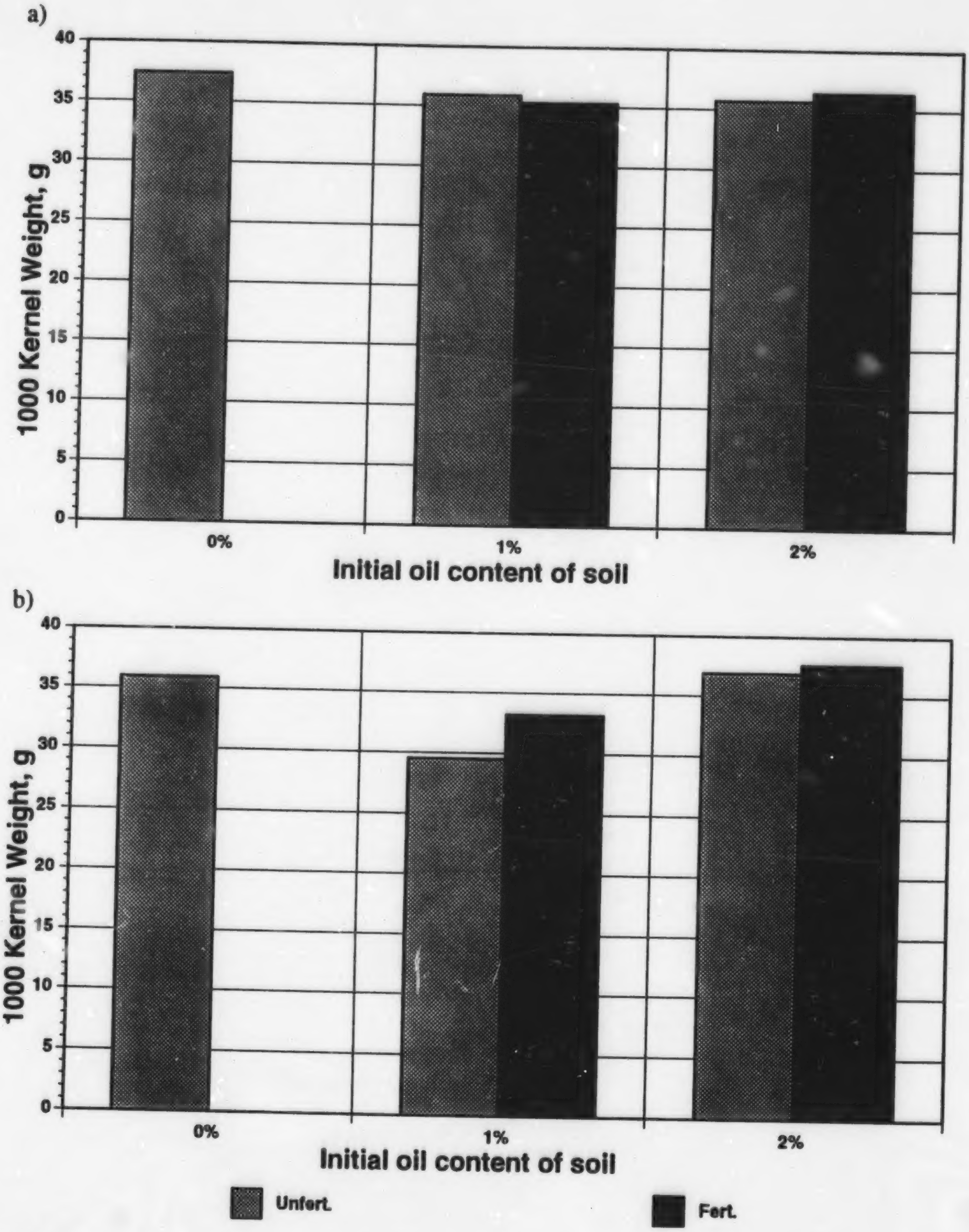


Figure 6.7-7. Effect of oily waste sludge incorporation and oil-related fertilization on 1000 kernel weight of a) durum, cv. Kyle, and b) oats, cv. Calibre, in 1999.

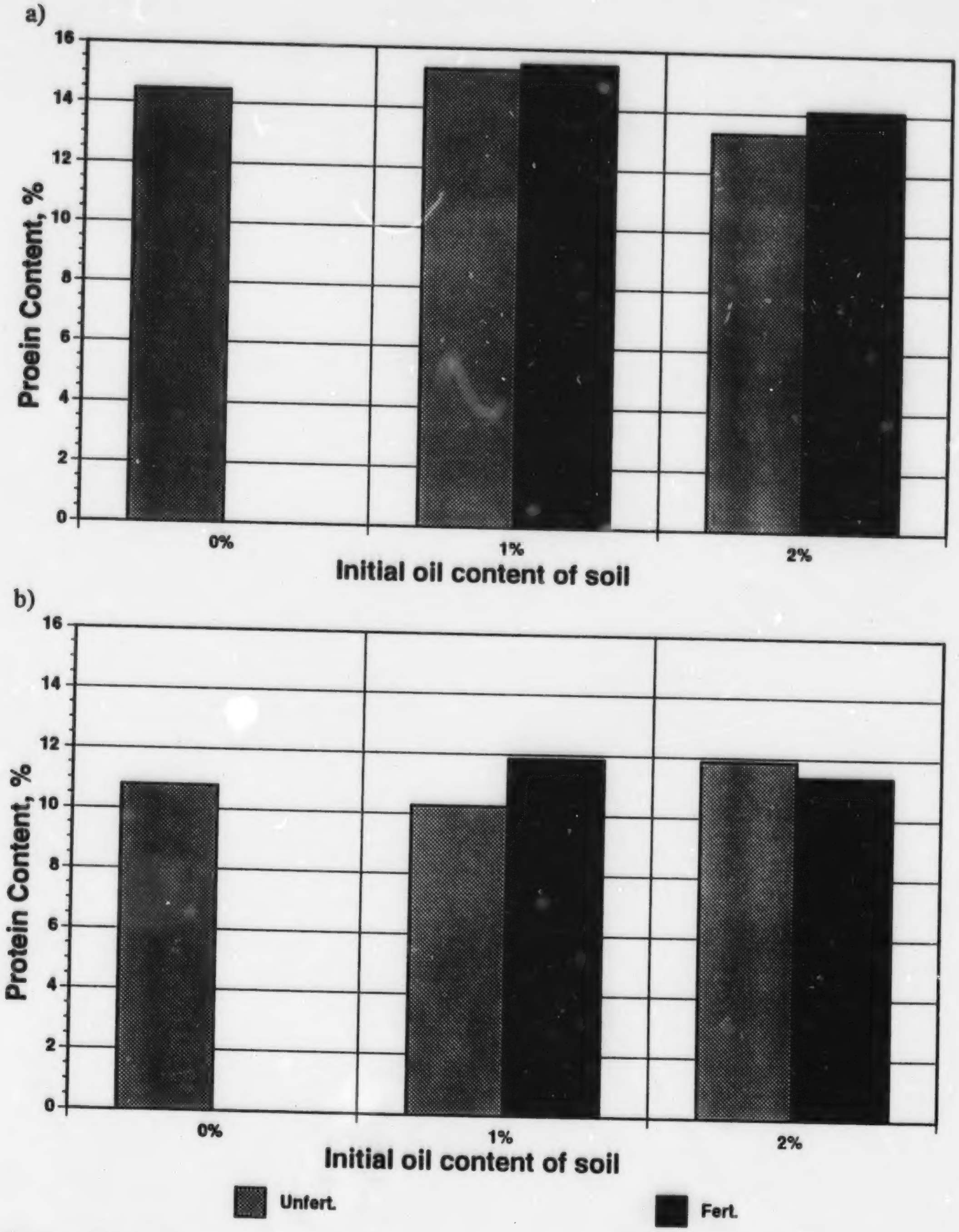


Figure 6.7-8. Effect of oily waste sludge incorporation and oil-related fertilization on protein content of a) durum, cv. Kyle, and b) oats, cv. Calibre, in 1999.

(Zeleny 1971) except oats for treatment O2F. The lack of reduction by sludge alone seems to indicate that there was adequate N available for both hydrocarbon bioconversion and plant growth at this early stage in the study. There was no consistent sludge or fertilizer effects associated with phosphorus content the grain of either crop (Figure 6.7-9).

Elemental analysis of the grain from O0U, O2F and O2U treatments was conducted for both crops. There were crop associated differences in the levels of several elements however, the results showed that there were no significant increases in the grain from either of the oiled treatments regardless of associated fertilization (data not shown). The levels of cadmium or lead were below detection limits for all treatments.

6.7.3 Nitrogen and phosphorus content in cereal straw and chaff

Oily waste sludge reduced the nitrogen content in the straw of both crops tested (Figure 6.7-10). This may reflect that that the crops are able to translocate more N from the tissues into the grain as the crop approaches maturity so that the 'seed' grain does is not affected by any nutrient deficiencies. Oil-related fertilization increased the N content of straw slightly for durum but in oat straw fertilization increased the N content well above the levels of the control especially the O2F treatment.

The effect of sludge or fertilization on phosphorus content of the crop residues was variable for both durum and oats (Figure 6.7-11). These results are similar to those found when corn was grown in plots with various amounts of used motor oil in a loamy sand that also showed that P content was not affected by the oil treatments (Giddens 1976).

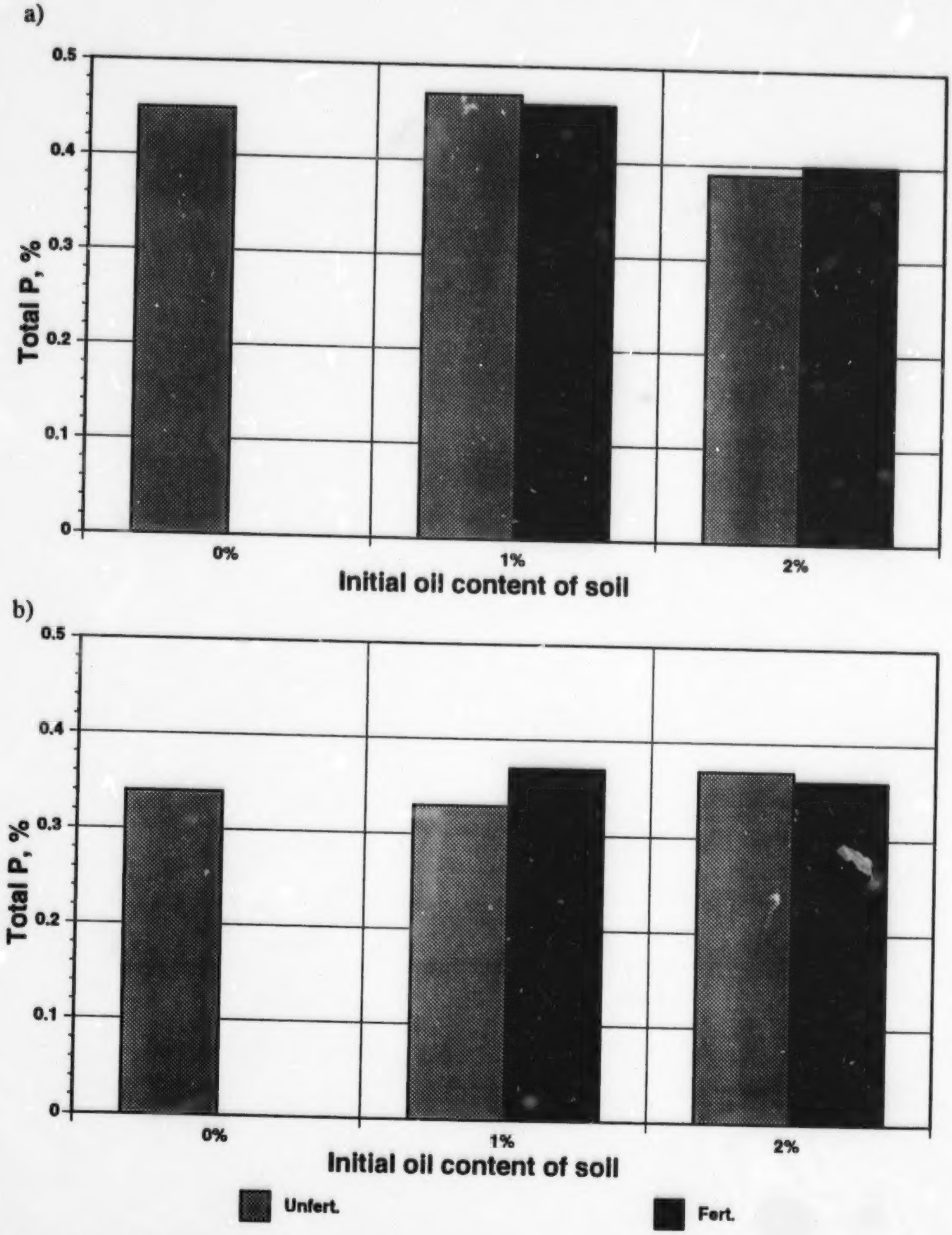


Figure 6.7-9. Effect of oily waste sludge incorporation and oil-related fertilization on the phosphorus content of grain from a) durum, cv. Kyle, and b) oats, cv. Calibre in 1999.

6.7.4 Conclusions – cereal yield and quality

Any detrimental effects, due to phytotoxicity of the hydrocarbons or other components, the oily waste sludge may have on crop production were not found in this study. It is possible that during the first field season that such effects were present but because of the overwhelming drought pressure at the site no confirmation of sludge associated effects could be attained.

In the second field season it became clear that any phytotoxicity that may have existed in the sludge had since been lost and yields were increased for all oily waste and fertilization treatments compared to checks. In addition to the high yields, there was little or no effect on nutrient content and most other quality traits were maintained.

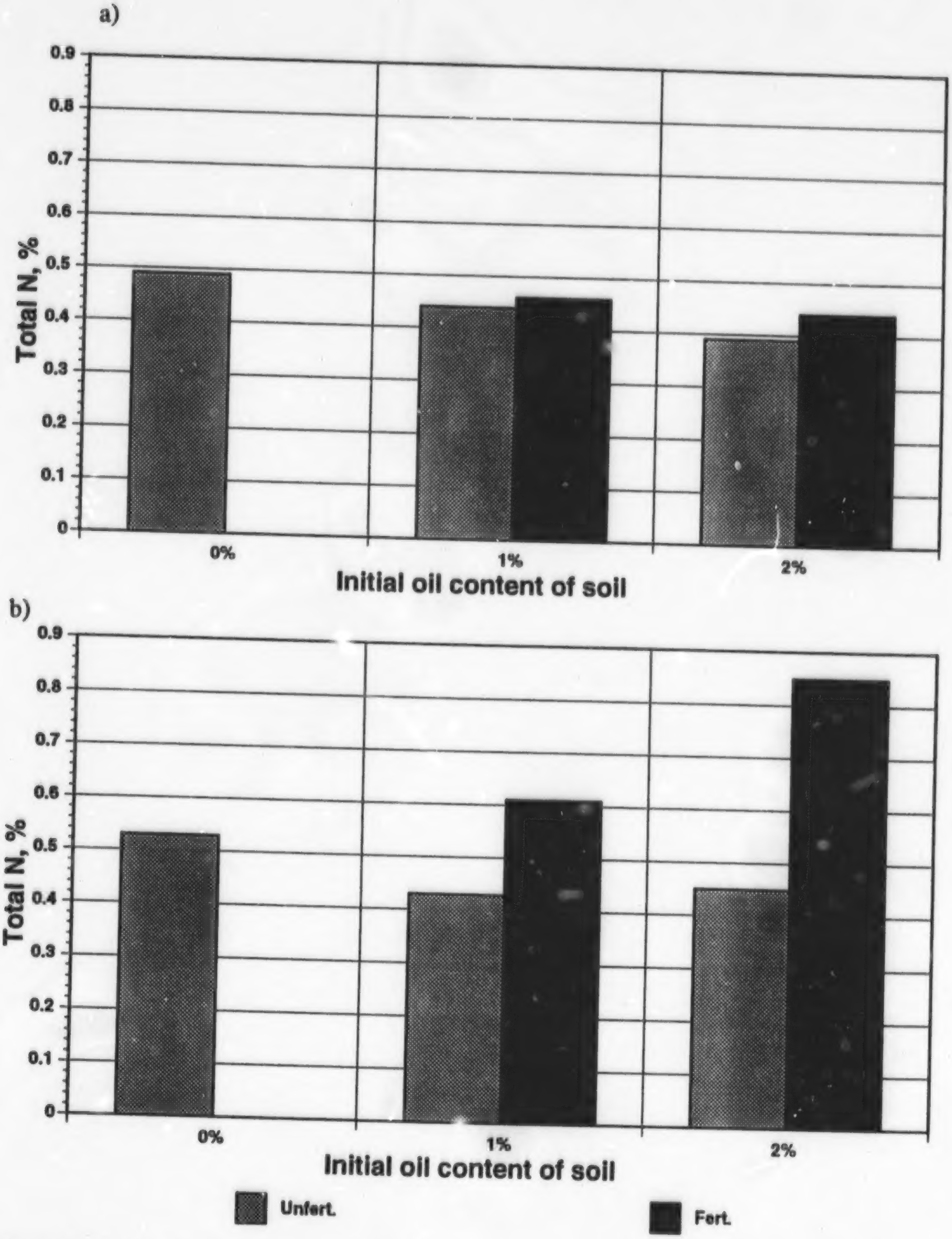


Figure 6.7-10. Effect of oily waste sludge incorporation and oil-related fertilization on the nitrogen content of straw from a) durum, cv. Kyle, and b) oats, cv. Calibre, in 1999.

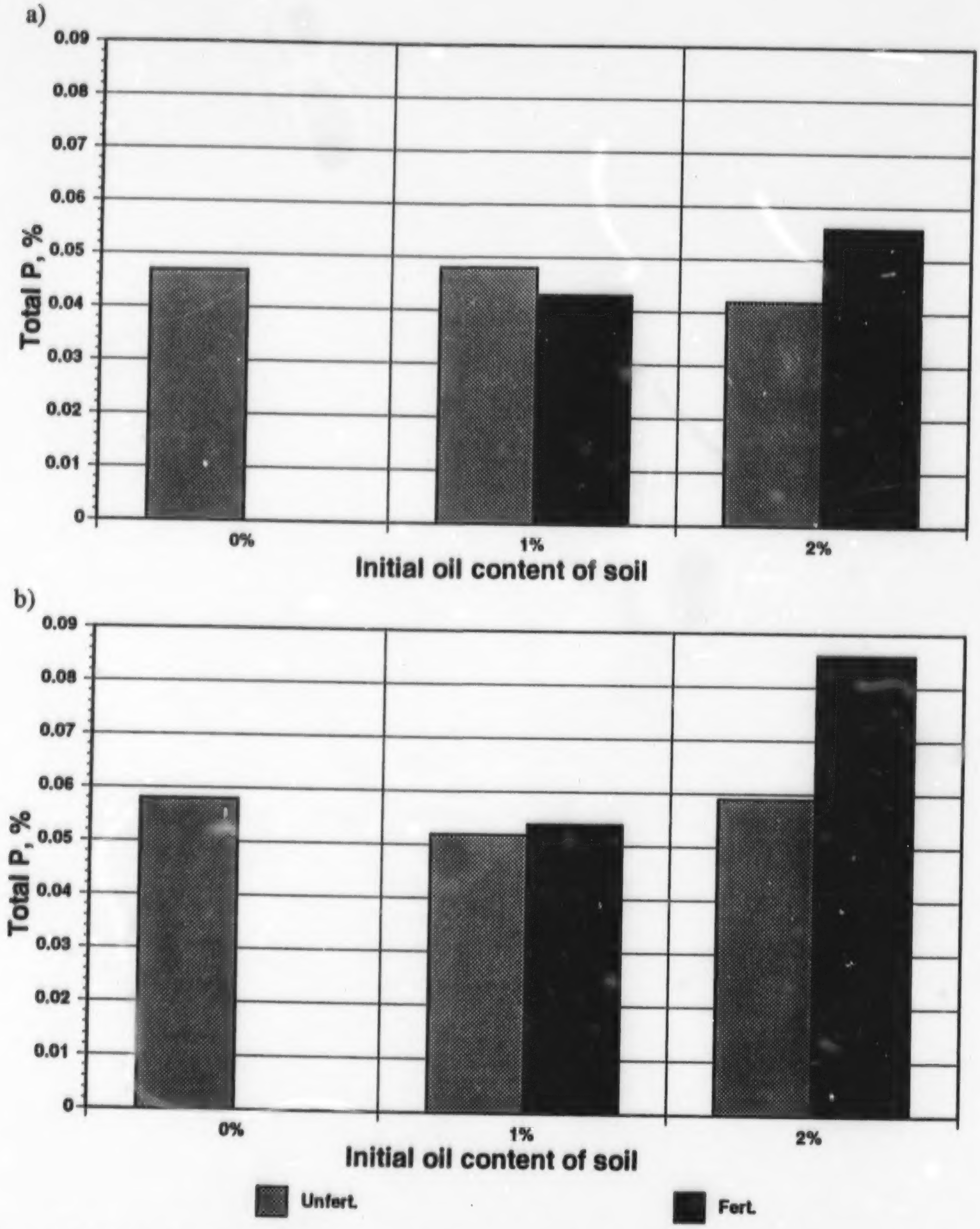


Figure 6.7-11. Effect of oily waste sludge incorporation and oil-related fertilization on the phosphorus content of straw from a) durum, cv. Kyle, and b) oats, cv. Calibre, in 1999.

6.8 GROUNDWATER QUALITY

Results from the first two groundwater sampling dates for all piezometers clearly showed there were no PAH or other hydrocarbon related organics detected in the groundwater (data not shown). The third sampling, in spring 2000, was taken to evaluate the groundwater for possible leached N and P from the heavy fertilizer applications in fall 1997 and spring 1998. The levels of nitrate-N were found to be higher than blank used as a control (Table 6.8-1). This confirms that nitrate was leached to significant soil depths and has reached the level of the groundwater. No difference was found between the blank and the piezometer samples for the levels of ammonium and phosphate.

Table 6.8-1. The level of nutrients detected in groundwater samples taken in spring 2000 at the study site near Richmound, SK.

Sample*	Nitrate-N	Ammonium-N	Phosphate-P
	------------------------------	ppm	------------------------------
Blank	0.02	0.05	0.01
P3	1.79	0.05	0.01
P4	26.24	0.05	0.00
P5	25.55	0.05	0.01
P6	9.82	0.05	0.01
P7	14.84	0.05	0.01

* The blank is distilled water that was taken to the field and used to wash the bailing tubes prior to water sampling. P2 was not sampled due to the presence of a dead mouse in the well.

7. RECOMMENDATIONS

As a result of this study, several recommendations can be made to improve the use of this technique as a means of disposing of heavy oil production and processing wastes in an environmentally sound and agriculturally beneficial manner.

1) During the incorporation of the oily waste sludge, more vigorous rotovation is essential to ensure thorough and uniform mixing of the sludge with the soil. This will help alleviate sampling errors and improve the ability to monitor oil biodegradation and bioconversion more accurately.
2) The addition of either 1% or 2% waste oil to this loamy sand resulted in great improvements to soil structure (reduced wind erodible fraction and increased waterstable aggregates) and crop production (yield increases for both durum and oats in the second field season with or without associated fertilization). This indicates that both levels of oily waste sludge incorporation are beneficial and depending on availability of waste and distance from the source either 1 or 2% oil can be used for amendment of marginal cropland.
3) Heavy fertilization is still deemed essential to provide the additional N & P required to promote bioconversion of the hydrocarbons into stable soil organic matter while limiting greenhouse gas emissions. Due to the extensive leaching of the applied nutrients observed in our study, it is very important to lower considerably the rates of fertilizer for each application. The overall amount of fertilizer applied should be reduced to about 2/3 of the levels we used because of dynamic recycling of nutrients through the soil microbial biomass during oil biodegradation. It will be critical to manage the fertilization so as to (i) initially, directly admix the nutrients to the hydrocarbons before or during the waste sludge application, and (ii) thereafter, apply the nutrients in many more

split applications at much lower rates and spaced out, in time, as suggested by the results from frquent sampling and analysis of the treated topsoil for availability of nutrients.

4) We believe the cropland that would receive the greatest benefit from application of this type of oily waste is land similar to that at our study site. This waste utilization technique should fit in well with conventional cropping practices in those areas with similar sandy dryland soils. We recommend that the oily waste be applied soon after harvest to a field that will then be entering a fallow phase. By applying the oily waste in early fall and not cropping the field for the next 19-20 months, any oily waste-associated phytotoxicity should be greatly diminished such that subsequent crop production will not be affected. The 'herbicidal' effect of the applied oily waste sludge should keep weeds sufficiently in check during this fallow phase that only one or two tillages would be required prior to the next field season. During the fallow year applications of fertilizer will be timely and can be worked into the soil with the weed control tillage operations. This will thoroughly mix the nutrients with the oiled soil layer while also aerating the soil to enhance microbial conversion of hydrocarbons into much-needed humus.

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SOILS

at

the

RICHMOUND
OILY WASTE
RESEARCH SITE

June 1998

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Semiarid Prairie Agricultural Research Centre
Agriculture and Agri-Food Canada
Research Branch

SOILS AT THE RICHMOUND OILY WASTE RESEARCH SITE

General Description

The oily waste research site is located on the SE quarter of Section 20, Tp 17, Rg 27, W3M in the RM of Fox Valley No. 171. It's in an area of hummocky morainal landscapes with gentle (2-5%) to moderate (5-10%) slopes. The actual site is located on the top of a large hill or upland area best described as a kame (a mound-like hill of ice-contact stratified drift). Although the soils in the immediate vicinity were mapped as Valor-Haverhill, Brown Chernozemic soils developed on shallow silty glacial lacustrine deposits overlying loamy glacial till (Valor), and loamy glacial till (Haverhill), the soils at the research site are much lighter (coarser) textured and are best described as belonging to the Hatton Soil Association (Ht). The Hatton Association consists chiefly of Chernozemic Brown soils developed on coarse to moderately coarse, weakly to moderately calcareous sandy glaciofluvial and lacustrine deposits. The deposits in this particular area are undoubtedly glaciofluvial and were probably deposited in contact with the glacier (ice-contact materials). There has obviously been some sorting of the glacial drift and collapse of the materials as the ice melted resulting in a very complex mixture of sand, glacial till and silty materials.

Most of the soils in the research area are fairly representative of the soils belonging to the Hatton Soil Association, especially those that have been mapped in complex with Haverhill soils. Hatton soils by definition contain less than 15% clay. The sixteen surface samples that were taken for mechanical analysis had on average 84% sand, 9.3% silt and 6.7% clay. In addition, all of the soils examined during the course of the survey also had very sandy surfaces. The Ap horizons were anywhere from 10 to 18 cm thick and were usually loamy sand in texture although a few sand and sandy loam textures were encountered. Figure 1 is a example of a typical Orthic Hatton profile found at the research site. The sandy deposits in this case were over 120 cm thick (the length of the hand auger). In many areas, however, the sandy deposits were fairly shallow (30-100 cm thick) and were underlain by glacial till as shown in Figure 2. The sandy deposits in this case were only 40 cm thick. Soils of this nature are referred to as Hatton over till (Ht/T). In some areas the sandy overlay was extremely shallow and consisted essentially of just the Ap horizon. In other areas there was only a thin layer of glacial till below the sandy surface. This layer of glacial till was in turn underlain by more sand. These latter two variations in the soil profiles are not typical of Hatton or Hatton over till soils. However, ice-contact materials can be very complex and quite variable. This is further illustrated by the fact that there were a few soil pits where the sandy Hatton materials were underlain by a thin layer of loamy or silty glaciolacustrine deposits (Figure 3). So instead of a thin layer of glacial till there was a thin layer of loamy or silty glaciolacustrine material sandwiched between the sand. The silty material shown in Figure 3 was approximately 17 cm thick and occurred anywhere from 16 to 40 cm below the surface at this particular point in the landscape.

Despite the relatively small size of the research site the slopes were quite variable, ranging from very gently sloping in south central portions along the western side of the research site to moderately and strongly sloping in northern and eastern portions of the research site. Due to the close proximity of glacial till in the area there were always a few stones on the surface in most areas but there were

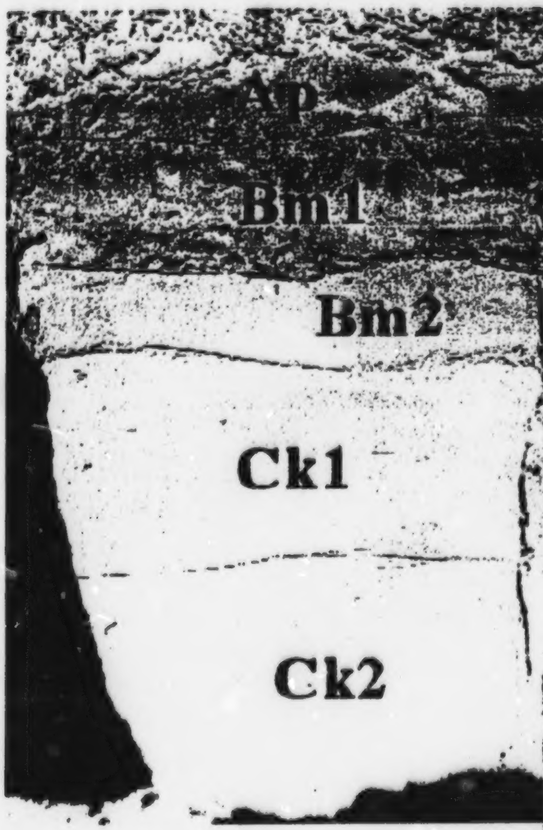
Ap
Bm1
Bm2
Ck1
Ck2

Figure 1. Orthic Hatton profile.

Ap	0 - 10 cm	(sand)
Bm1	10 - 20 cm	(sand)
Bm2	20 - 34 cm	(sand)
Ck1	34 - 54 cm	(sand)
Ck2	54 - 100 cm	(sand)

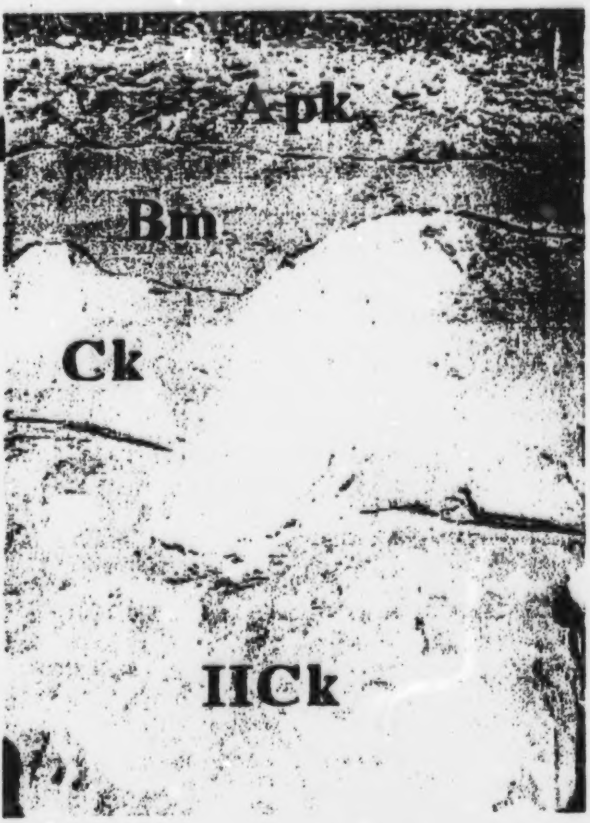
Apk
Bm
Ck
IICk

Figure 2. Orthic Hatton profile over till.

Apk	0 -9 cm	(sand)
Bm	9 - 18 cm	(sand)
Ck	18 - 40 cm	(sand)
IICk	40 - 100 cm	(till)

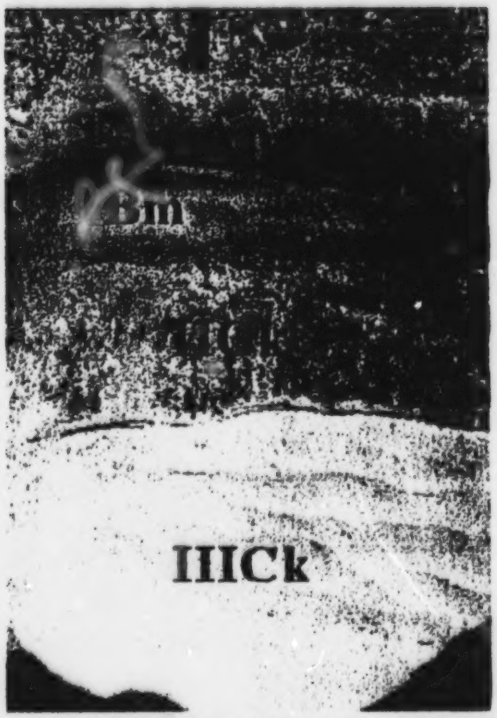

Figure 3. Orthic Hatton profile with a layer of silty material within the sand.

Ap	0 - 11 cm	(sand)
Bm	11 - 21 cm	(sand)
IICk	21 - 38 cm	(silty material)
IIICk	38 - 100 cm	(sand)

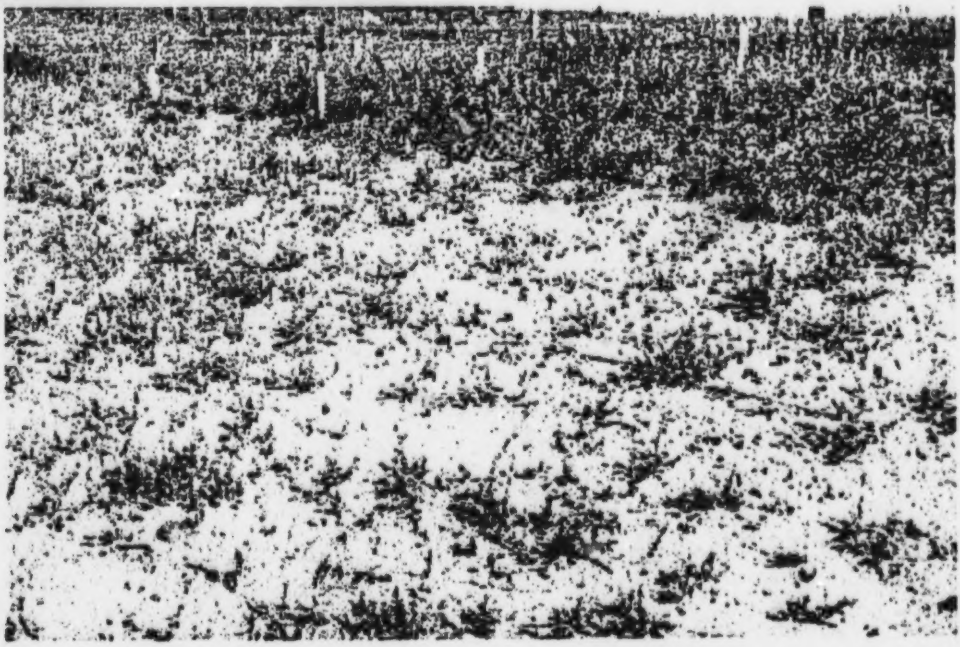

Figure 4. Soil surface at the research plot showing the stones and small pebbles left behind as a result of wind erosion.

never enough to seriously hinder cultivation. Wind erosion, however, appears to have been a problem. It was evident that materials were being eroded from west-central portions of the plot and deposited onto various areas in the eastern half of the research site. The small stones and pebbles left on the surface in Figure 4 shows that wind erosion has removed much of the finer material. The layering or bedding observed in the soil profiles at several points in eastern portions of the research site also provided evidence for the accumulation of eroded material. The steeper slopes in northern and eastern portions of the site also make many of these areas susceptible to water erosion.

The soil profiles encountered during the course of the survey consisted of an equal number of Orthic Brown Chernozems and a mixture of Rego and Calcareous Brown Chernozems. There were however, three times as many rego profiles as there were calcareous profiles. Many of the orthic profiles were carbonated at the surface but had lime-free Bm horizons. The carbonate present in the surface horizons was probably the result of erosion from carbonated upper slope positions or the result of mixing brought about by cultivation. The profile distribution appeared to be rather random throughout the research site.

The Soil Survey

The initial soil survey was carried out on August 26 and 27, 1997. The area at that time consisted of two 50 m x 100 m plots, one in a summerfallow field and the other in the immediately adjacent cropped field, separated by an 8 m wide service area between the two plots. Soil pits were dug along the outer edges and through the middle of each plot running from south to north at 20 m intervals resulting in 36 soil pits. The pits were dug by hand with a spade to approximately 60 cm in depth in order to identify the profile and surficial deposits and then augered to approximately 120 cm to check on the uniformity of the surficial deposits within the control section. Hand texturing was used to estimate the percentages of sand, silt and clay.

In order to allow for additional replication the research plot area was extended another 34 m to the north. A survey of this additional area was completed on October 27, 1997. Instead of being 50 m wide the additional areas were only 44 m wide. Soil inspections were again made along the outer edges of the two adjacent fields and through the middle at 17 m intervals in the south to north direction resulting in an additional 6 inspection sites per field for a total of 12 additional inspection sites (48 soil pits overall). Five areas were delineated on the soil map (Fig. 5). The following is an example of the symbols used on the map and in the text along with an explanation of the individual components of the map symbol. The location of the soil inspection sites and individual research plots within the surveyed area is illustrated in Figure 6.

Sequence of Map Symbols

Association and Map Unit : Surface Texture   e.g. HtA/T : sl-ls
Slope Class and Surface Expression   3h

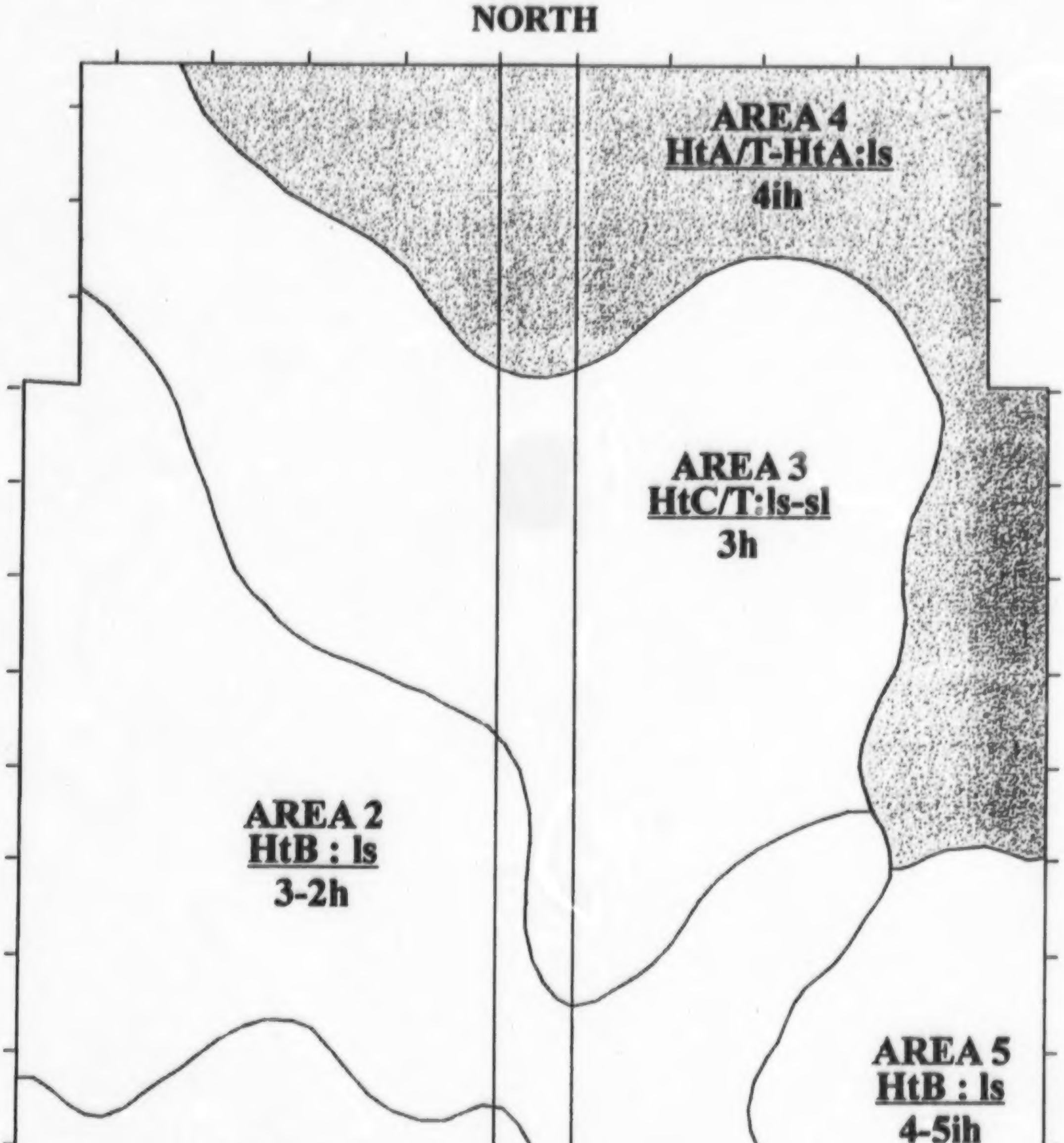
NORTH
AREA 4
HtA/T-HtA:ls
4ih
AREA 3
HtC/T:ls-sl
3h
AREA 2
HtB : ls
3-2h
AREA 5
HtB : ls
4-5ih
AREA 1
HtC/T : sl-ls
3h
Scale 1:700
10m

Figure 5. Soil map of the Richmound oily waste research site.

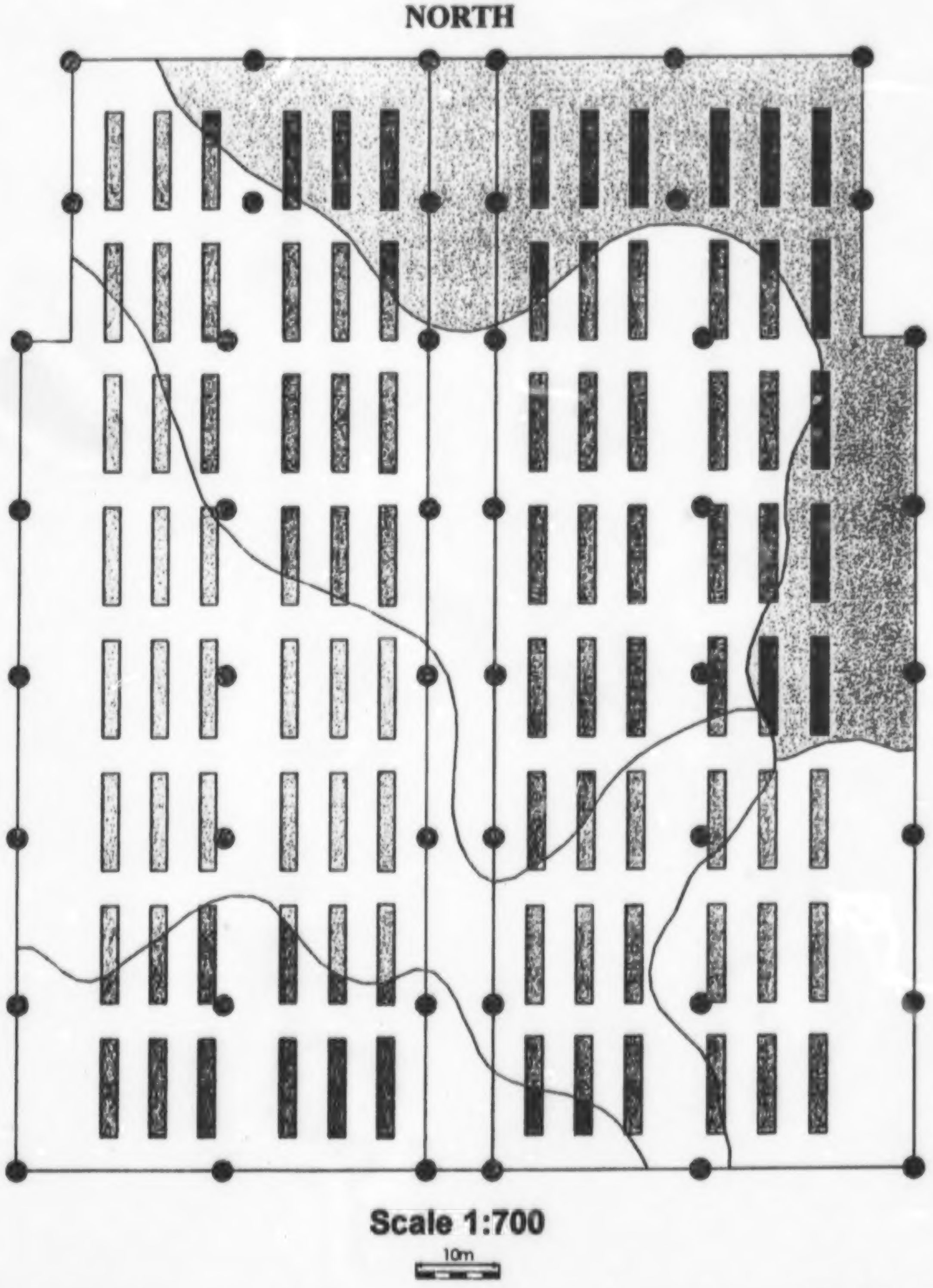
NORTH
Scale 1:700
10m

Figure 6. Soil inspection points (●) and location of field plots.